Gebrauchsanleitung

Titrator
TitroLine *KF*

Operating Instructions

Titrator
TitroLine *KF*

Mode d'emploi

Titrateur
TitroLine *KF*

Manual de instrucciones

Titulador
TitroLine *KF*

SCHOTT Instruments

Gebrauchsanleitung ..... Seite 1 .. 38

Wichtige Hinweise: Die Gebrauchsanleitung vor der ersten Inbetriebnahme des Titrators TitroLine *KF* bitte sorgfältig lesen und beachten. Aus Sicherheitsgründen darf der Titrator TitroLine *KF* ausschließlich nur für die in dieser Gebrauchsanleitung beschriebenen Zwecke eingesetzt werden.

Bitte beachten Sie auch die Gebrauchsanleitungen für die anzuschließenden Geräte.

Alle in dieser Gebrauchsanleitung enthaltenen Angaben sind zum Zeitpunkt der Drucklegung gültige Daten. Es können jedoch von SCHOTT sowohl aus technischen und kaufmännischen Gründen, als auch aus der Notwendigkeit heraus, gesetzliche Bestimmungen der verschiedenen Länder zu berücksichtigen, Ergänzungen am Titrator TitroLine *KF* vorgenommen werden, ohne dass die beschriebenen Eigenschaften beeinflusst werden.

Operating Instructions ..... Page 39 ... 76

Important notes: Before initial operation of the Titrator TitroLine *KF* please read and observe carefully the operating instructions. For safety reasons the Titrator TitroLine F*K* may only be used for the purposes described in these present operating instructions.

Please also observe the operating instructions for the units to be connected.

All specifications in this instruction manual are guidance values which are valid at the time of printing. However, for technical or commercial reasons or in the necessity to comply with the statuary stipulations of various countries, SCHOTT may perform additions to the Titrator TitroLine *KF* without changing the described properties.

Mode d’emploi ..... Page 77 .. 114

Instructions importantes: Prière de lire et d’observer attentivement le mode d'emploi avant la première mise en marche du Titrateur TitroLine *KF*. Pour des raisons de sécurité, le Titrateur TitroLine *KF* pourra être utilisé exclusivement pour les usages décrits dans ce présent mode d'emploi.

Nous vous prions de respecter également les modes d'emploi pour les appareils à connecter.

Toutes les indications comprises dans ce mode d’emploi sont données à titre indicatif au moment de l'impression. Pour des raisons techniques et/ou commerciales ainsi qu'en raison des dispositions légales existantes dans les différents pays, SCHOTT se réserve le droit d'effectuer des suppléments concernant le TitrateurTitroLine *KF* qui n’influencent pas les caractéristiques décrits.

Manual de instrucciones ..... Página 115 ... 152

Instrucciones importantes: Primeramente, lean y observen atentamente el manual de instrucciones antes de la primera puesta en marcha del Titulador TitroLine *KF*. Por razones de seguridad, el Titulador TitroLine *KF* sólo debe ser empleada para los objetivos descritos en este manual de instrucciones.

Por favor, respeten las indicaciones descritas en los manuales de instrucciones de los equipos antes de conectarlos.

Todos los datos contenidos en este manual de instrucciones son datos orientativos que están en vigor en el momento de la impresión. Por motivos técnicos y / o comerciales, así como por la necesidad de respetar normas legales existentes en los diferentes países, SCHOTT puede efectuar modificaciones concernientes al Titulador TitroLine *KF* sin cambiar las características descritas.

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1 Properties of the TitroLine *KF* Titrator

1.1 General

Note on disclaimer of liability

The measurement or analysis results depend on a multitude of factors. So please verify the plausibilty of the measurement or analysis results on a regular basis, accompanied by appropriate reliability tests. In this regard, please adhere to the usual validation methods.

The TitroLine *KF* Titrator is a very compact, integral volumetric titrator for the determination of the water contents according to Karl Fischer.

The accurately working dosing system meets the most important prerequisite for correct titration. In addition to the high-precision glass cylinder made of DURAN® borosilicate-glass, the play-free motor spindle ensures exact titration results.

The very simple operator prompting allows you uncomplicated working. The display will ask you to select the respective function or to set the corresponding parameters. Even during the execution of a function the display of the titrator will keep you at any time informed about its present status.

The methods for sample titration, the titer and blank-value determination are pre-parameterised, so that titration can begin immediately upon the set-up. The installed titration algorithm adapts automatically to the water contents of the sample. As a rule, only few samples require any modification in of the titration parameters.

Since almost all parts are pre-mounted, the measurement rig can be set up easily in less than 10 minutes. Using the starter kit coming with the system, you may start to work immediately - as a rule with a titer determination.

A connected TZ 2825 PC mini keyboard facilitates the input of the weighed-in sample quantity and of an alphanumeric sample designation.

1.2 Titration solutions

Solutions to be used:

Any modern Karl-Fischer reagents known so far can be used.

General rules:

The respectively applicable safety guidelines on handling chemicals are to be observed under all circumstances. This applies in particular to inflammable and/or etching liquids.

Notes: When used in this text, the following signs have the following meaning:

⚠ = note on possible danger
<< ... >> = contents of the display
< ..... > = key
📖 = please refer to, for instance, figure, chapter, operating instructions

KONFORMITÄTSERKLÄRUNG
DECLARATION OF CONFORMITY
DÉCLARATION DE CONFORMITÉ

Wir erklären in alleiniger Verantwortung, dass das Produkt

We declare under our sole responsibility that the product

Nous déclarons sous notre seule responsabilité que le produit

Titrator
TitroLine *KF*

mit

Titrierstand
TM KF

Titrator
TitroLine *KF*

with

Titration Stand
TM KF

Titrateur
TitroLine *KF*

avec

Support de titration
TM KF

auf die sich diese Erklärung bezieht, übereinstimmt mit den Normen

to which this declaration relates is in conformity with the standards

DIN 12 650, Teil 5

und / and / et

EN ISO 8655-3

auquel se réfère cette déclaration est conforme aux normes

und mit den normativen Dokumenten

and the normative documents

et aux documents normatifs

Technische Daten

Titrator TitroLine *KF*
01.02.2000

und / and / et

Titrierstand TM KF
01.02.2000

SCHOTT Instruments GmbH
Hattenbergstraße 10
55122 Mainz
Deutschland, Germany, Allemagne

22. Juli, July 22nd, 22 Juillet 2002
AGQSF 0000-A063-02/040802

Translation of the legally binding German version

Technical data TitroLine *KF* (basic device)

State 01.02.2000

Conformity:	DIN 12 650, Part 5, conformity sign
CE sign: CE	EMC compatibility according to the Council Directive 89/336/EWG; Generic emission according to EN 50 081, Part 1 Generic immunity according to EN 50 082, Part 2 Low-voltage directive according to the Council Directive 73/23/EWG, last modified by the Council Directive 93/68/EWG Testing basis EN 61 010, Part 1
Country of origin:	Deutschland / Made in Germany
Dosing unit:	
Cylinder:	20 ml DURAN® (borosilicate glass)
UV protection:	Protective coating made of Ultem 1000, colour: amber
Valve:	Motor-driven 3/2-way valve made of PTFE / ETFE
Hoses:	FEP with UV protection
Dosing accuracy:	according to EN ISO 8655-3: maximum permissible systematic error ± 0.2 maximum permissible random error ± 0.07
Display:	Matrix LCD display 69 x 39 mm, 64 x 128 pixels with background lighting, contrast can be set using a knurled wheel
Connections:	
Electrode:	Connection for double platinum electrode. Output voltage 100 mV Adjustable by software between 5 – 200 mV. Connection: sockets 2 x 4 mm Measuring range 0 – 99.9 µA, display resolution: 0.1 µA
Keyboard:	5-channel DIN socket for TZ 2825
Stirrer:	Plug connection with integrated low-voltage supply (15 V =) incorporated in the bottom of the casing of the titrator for TM 96 Stirrer
TM KF Titration Stand:	Socket for low-voltage connection -jack plug-, positive pole on pin contact, internal contact ∅ = 2.1 mm, USA/Japan; low-voltage output 12 V / –
RS-232-C-interfaces:	two bi-directional RS-232-C interfaces Plug connections: 4-channel round plug mini DIN Configuration of the RS-232-C interfaces 1 and 2 separate configuration Baud: 1200, 2400, 4800 or 9600 baud; Word length: 7 or 8; stop bit 1, parity: no, even or odd; address: 00 ... 15
Power supply:	corresponds to protective system II according to DIN EN 61 010, Part 1, not suitable for use in environment with explosion hazard Mains: 230 V~, 50 / 60 Hz or 115 V~; 50 / 60 Hz Power draw: 30 VA
Materials:	
Casing :	Polypropylene
Front foil:	Polyester
Casing dimensions:	310 x 135 x 205 mm (w x h x d), height incl. dosing unit, without Titration Stand 310 x 265 x 205 mm (w x h x d) with TM KF Titration Stand and titration vessel
Weight:	approx. 2.1 kg for basic device, about 3.2 kg for complete device with Titration Stand
Ambient conditions:	Ambient temperature: + 10 ... + 40 °C for operation and storage humidity according to EN 61 010, Part 1: max. relative humidity 80 % for temperatures up to 31 °C, linear decrease down to 50 % relative humidity at a temperature of 40 °C

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Translation of the legally binding German version

Technical data **TM KF Titration Stand** in connection with the **TitroLine *KF* Titrator** State 01.02.2000

CE sign: CE	EMC compatibility according to the Council Directive 89/336/EWG; Generic emission according to EN 50 081, Part 1 Generic immunity according to EN 50 082, Part 2 Low-voltage directive according to the Council Directive 73/23/EWG, last modified by the Council Directive 93/68/EWG Testing basis EN 61 010, Part 1
Country of origin:	Made in Germany
Pump:	Free volume flow - air-: pumping capacity 2.25 l / min pumping pressure max. 1.5 bar Pumping capacity liquid media approx. 0.8 l / min
Stirring speed:	50 ... 1000 U/min
Hoses:	PVC hose, outside diameter 6 x 1 mm PTFE hose, outside diameter 4 x 0.5 mm
Connections	
Power supply:	12 V low-voltage input / – on the back panel of the titration stand (top); plug connection: socket for low-voltage connection -jack plug-, positive pole on pin contact, internal contact ∅ = 2.1 mm, USA/Japan, power supply via TitroLine *KF* Titrator
Casing	
Material :	Polypropylene;
Dimensions:	approx. 78 x 130 x 180 mm, h x w x d (height excl. stand rod)
Weight:	approx. 0.9 kg
Environment:	not suitable for use in hazardous environment!
Climate:	Ambient temperature: + 10 °C ... + 40 °C for storage and transportation air humidity: according to EN 61 010, Part 1: maximal relative humidity 80 % for temperatures up to 31 °C, linear decrease down to 50 % relative humidity at a temperature of 40 °C

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2 Warning and safety information

The TitroLine *KF* Titrator corresponds to protection class II. It was manufactured and tested according to DIN EN 61 010, Part 1, Protective Measures for Electronic Measurement Devices and has left the factory in an impeccable condition as concerns safety technology. In order to maintain this condition and to ensure safe operation, the user should observe the notes and warning information contained in the present operating instructions. Development and production is done within a system which meets the requirements laid down in the DIN EN ISO 9001 standard.

For reasons of safety, the TitroLine *KF* Titrator must be opened by authorised persons only; this means, for instance, that work on electrical equipment must only be performed by qualified specialists.

⚠ **In the case of nonobservance of these provisions the TitroLine *KF* Titrator may constitute a danger: electrical accidents of persons or fire hazard. Moreover, in the case of unauthorised intervention in the TitroLine *KF* Titrator as well as in the case of negligently or deliberately caused damage, the warranty will become void.** ⚠

Prior to switching the device on it has to be ensured that the operating voltage of the TitroLine *KF* Titrator matches the mains voltage. The operating voltage is indicated on the specification plate. Nonobservance of this provision may result in damage to the TitroLine *KF* Titrator or in personal injury or damage to property.

Prior to switching the device on it has to be ensured that the operating voltage of TitroLine *KF* Titrator matches the mains voltage. The operating voltage is indicated on the specification plate. Nonobservance of this provision may result in damage to the TitroLine *KF* Titrator or in personal injury or damage to property.

Examples for the assumption that a safe operation is no longer possible
- ➲ the package is damaged,
- ➲ the TitroLine *KF* Titrator shows visible damages,
- ➲ the TitroLine *KF* Titrator does not function properly,
- ➲ liquid has penetrated into the casing,
- ➲ the TitroLine *KF* Titrator was subjected to technical modifications or to interventions in the form of attempted repair work by unauthorised personnel.

In the case of deviations from the intended proper use of the device, it is up to the user to evaluate the occurring risks.

The TitroLine *KF* Titrator must not be stored or operated in humid rooms.

For reasons of safety, the TitroLine *KF* Titrator must only be used for the range of application described in the present operating instructions.

In the case of deviations from the intended proper use of the device, it is up to the user to evaluate the occurring risks.

⚠ **The relevant regulations regarding the handling of the substances used have to be observed:** The Decree on Hazardous Matters, the Chemicals Act, and the rules and information of the chemicals trade. It has to be ensured on the side of the user that the persons entrusted with the use of the TitroLine *KF* Titrator are experts in the handling of substances used in the environment and in the TitroLine *KF* Titrator, or that they are supervised by specialised persons, respectively.

During all work with titration solutions: ⚠ **Please wear protective glasses!** ⚠

When the piston moves upwards within the cylinder, owing to the design of the system, a microfilm of dosing liquid or titration solution will always remain adhered to the inner wall of the cylinder, but this has no influence on the dosing accuracy. This small residue of liquid, however, may evaporate and thus penetrate into the zone underneath the piston, and if inadmitted liquids are being used, the materials of the TitroLine *KF* Titrator may be dissolved or corroded (please refer also to "Maintenance and Care").

The TitroLine *KF* Titrator is equipped with integrated circuits (EPROMs). X rays or other high-energy radiation may penetrate through the device’s casing and delete the program.

3 Setting up and commissioning

3.1 Unpacking and setting up of the TitroLine *KF* Titrator

All the components of the TitroLine *KF* Titrator are contained within one single packing and arranged in such a manner that they can be taken out very easily.

Please note that also all the small accessories are taken out of the packing. For the scope of delivery, please refer to the packing list.

First, take TitroLine *KF* basic device out of the packing.

Set up the TitroLine *KF* Titrator at any flat surface, and connect the mains plug to the mains socket. Prior to plugging in the mains plug it has to be ensured that the operating voltage of the TitroLine *KF* Titrator matches that of the the mains voltage. The operating voltage is indicated on the specification plate on the bottom of the TitroLine *KF* Titrator.

⚠ The TitroLine *KF* Titrator is not suitable for use in explosive-hazardous environments. ⚠

3.2 Installation of the TM KF Titration Stand and of the titration vessel

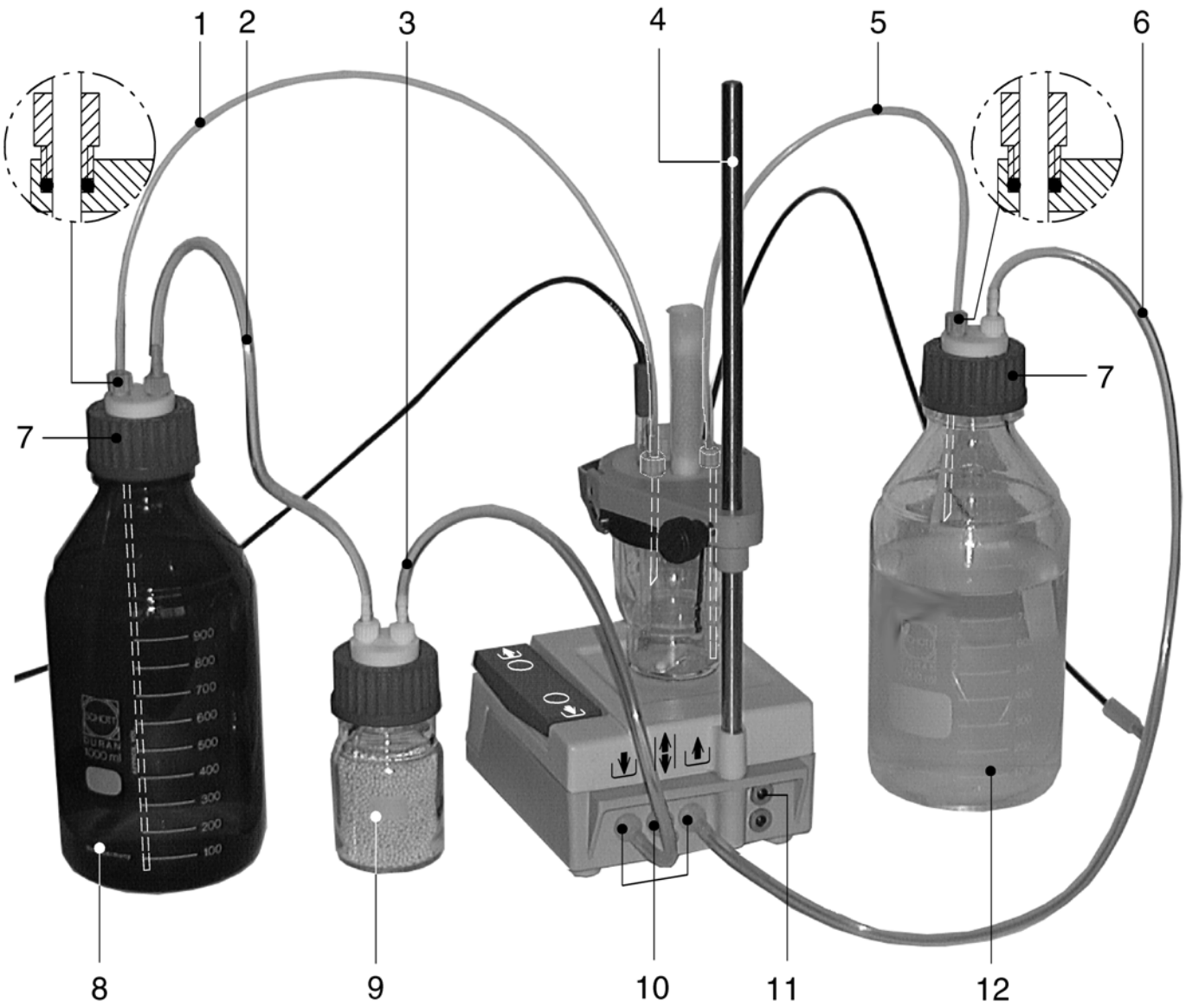
As a rule, you will place the titration rig directly to the right of the TitroLine *KF* Titrator.

Seen from the front:

- Connect the right hose olive located at the back panel of the titration rig and the hose olive on the desiccation bottle (**figure 2**, item 9) using one of the short PVC hoses (**figure 2**, item 10 and item 3).
- Connect the other hose olive of the desiccation bottle to the hose olive of one of the square bottles (solvent bottle) using the other short PVC hose (refer to **figure 2**, item 2).
- Connect the left hose olive at the backside of the titration rig and the hose olive on the waste bottle using the longer PVC hose (**figure 2**, item 10 and 6).
- Insert the stand rod (**figure 2**, item 4) into the corresponding holder on the titration rig, then tighten it manually.
- Use the knurled screw to fasten the titration vessel to the stand rod in such a manner that the bottom of the titration vessel sits directly on the upper surface of the titration rig.
- Adjust the dosing and waste hoses as is shown in **figure 2**. This applies to the titration vessel, the storage bottle, and the waste bottle. Subsequently, tighten the screwed connections with the hoses on the titration vessel and on the bottles.
- ***)** Fill the desiccation bottle (**figure 2**, item 9) up to two thirds with the enclosed 0.3 nm molecular sieve.
- Also, fill some molecular sieve into the desiccation pipe for the titration vessels. First, you should put some of the enclosed cotton into the desiccation pipe, so no molecular sieve will fall into the titration vessel.
- Fill the solvent into the 1 l solvent bottle. Instead of the square laboratory bottles coming with the device, it is also possible to use other reagents bottles with a GL 45 or S 40 (TZ 1795 adapter) thread.
- If you wish to inject liquids through a septum, unscrew the large screwed connector, and then put one of the enclosed septums into the opening. You can remove the black O-ring before. Subsequently, tighten the screw connector manually.
- Screw the titration hose onto the valve of the titrator.

All the other hose connectors as well as the threaded connections of the TZ 1770 or TZ 1772 titration vessel are already pre-mounted.

Figure 2 Hose connections (seen from behind)



1 PTFE filling hose:
Storage bottle ⇔ titration vessel

2 PVC hose for overpressure in the storage bottle:
Storage bottle ⇔ receiver drier

3 PVC hose for overpressure in the storage bottle:
Receiver drier ⇔ titration vessel

4 Stand rod

5 PTFE suction hose:
Titration stand ⇔ waste bottle

6 PVC hose for vacuum in the waste bottle:
Titration stand ⇔ waste bottle

7 GL 45 screwed connections with adapter for hose connections and screw connections

8 Storage bottle 1 l, brown

9 Receiver drier 100 ml
***)** see previos page

10 Olives for connection of the PVC hoses

11 Mains-unit connector
U = 12 V-

12 Waste bottle 1 l, bright

Power supply of the TM KF Titration Stand

The power supply to the titration rig comes from the titrator. Connect the titration stand to the titrator using the enclosed low-voltage cable (please refer to **figure 2**, item 11 and **figure 4**, 12 V DC).

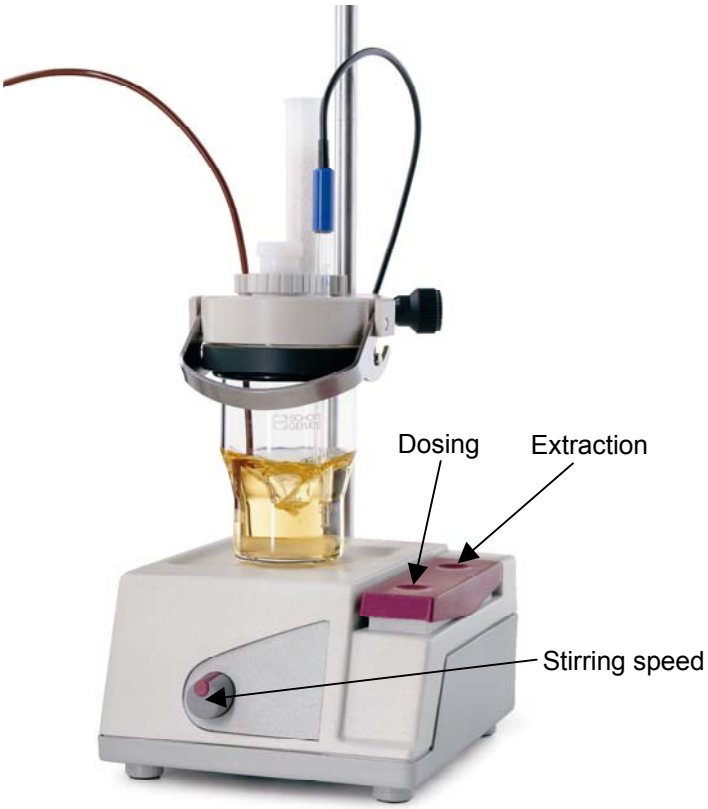
Working with the titration stand

- Dosing: Press the front part of the key rocker to pump solvent into the titration vessel. Additive dosing will continue as long as you press the key.
- Dosing: Press the front part of the key rocker to pump solvent into the titration vessel. Additive dosing will continue as long as you press the key.

Note: Please observe the filling level of the waste bottle. Prior to extracting the finish-titrated solution, please ensure that the waste bottle can accommodate at least this quantity of solution.

The built-in magnetic stirrer (stirrer for magnetic stirrer rod) will stir the liquids in the titration vessel. Use the turning knob on the front panel of the titration stand to set the stirring speed (**figure 3**).

Figure 3



Malfunction

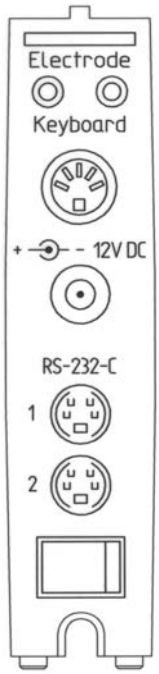
Problems might occur if the hoses were connected improperly, or if the pressure or vacuum systems should contain a leaking screw connection. This will lead to a cessation of the solvent discharge after only a few seconds after the start of the pump. So when checking the hoses, please pay particular attention to the tightness between the flanged connections and their adapters. The same applies to the hose connections of the receiver drier or the interchangeable desiccating tube unit.

Note: Wrinkled hoses will cause dosing and extraction problems. The tightness of the hoses including their connections should be checked on a regular basis. Replace defective items if necessary.

If solvent should coast out of the storage bottle during additive dosing, please place the bottle on a level below that of the titration vessel. Likewise, if solvent should coast out of the storage bottle during the change from dosing to extraction, you should wait for a few seconds during the change.

3.3 Connecting the PC mini keyboard and the electrode

Figure 4 Connectors on the back panel of the TitroLine *KF* Titrator



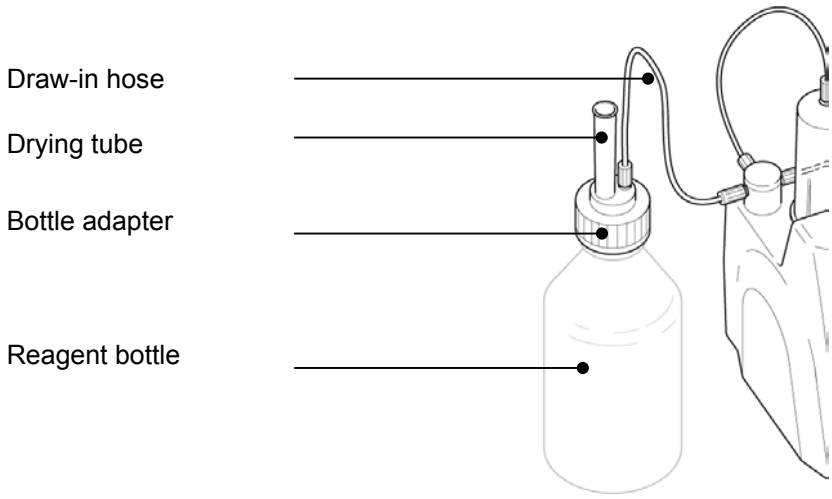
Top down:

- Electrode: TZ 1106 micro double platinum electrode connector
- Keyboard: TZ 2825 PC mini keyboard connector, please refer to **figure 7**
- 12 V DC: TM KF Titration Stand connector; power supply 12 V
- RS-232-C 1: Printer / PC
- RS-232-C 2: Balance and connection of additional titrators,
  (📖 **Chapter 7.2** Daisy chaining of several TitroLine *KF* Titrators → Daisy-Chain
- Mains switch

3.4 Connecting the reagents bottles (titrant, titration agents)

The scope of delivery of the TitroLine *KF* Titrator also includes a GL-45 adapter as well as a 1 l brown-glass bottle. Please insert the extraction hose of the TitroLine *KF* Titrator through the small opening of the adapter (**figure 5**) so that the hose immerses deeply enough into the reagent bottle. Put some of the cotton into the desiccation pipe (**figure 5**), fill it with the molecular sieve.

Figure 5 Connecting the reagent bottle to the TitroLine *KF* Titrator



You can now fill the brown reagent bottle with the titration agent.

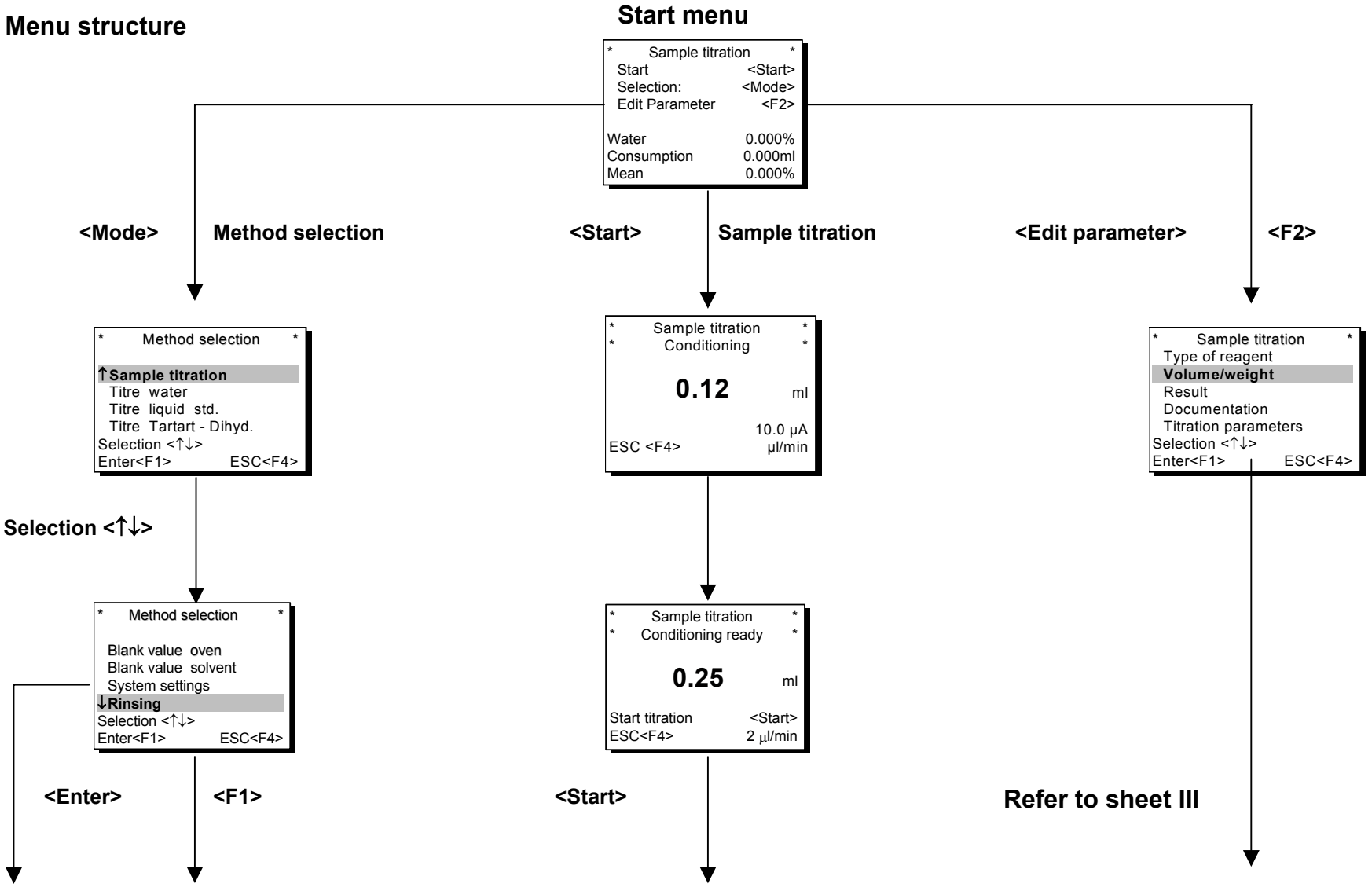
⚠ **Please use a funnel for filling.** ⚠

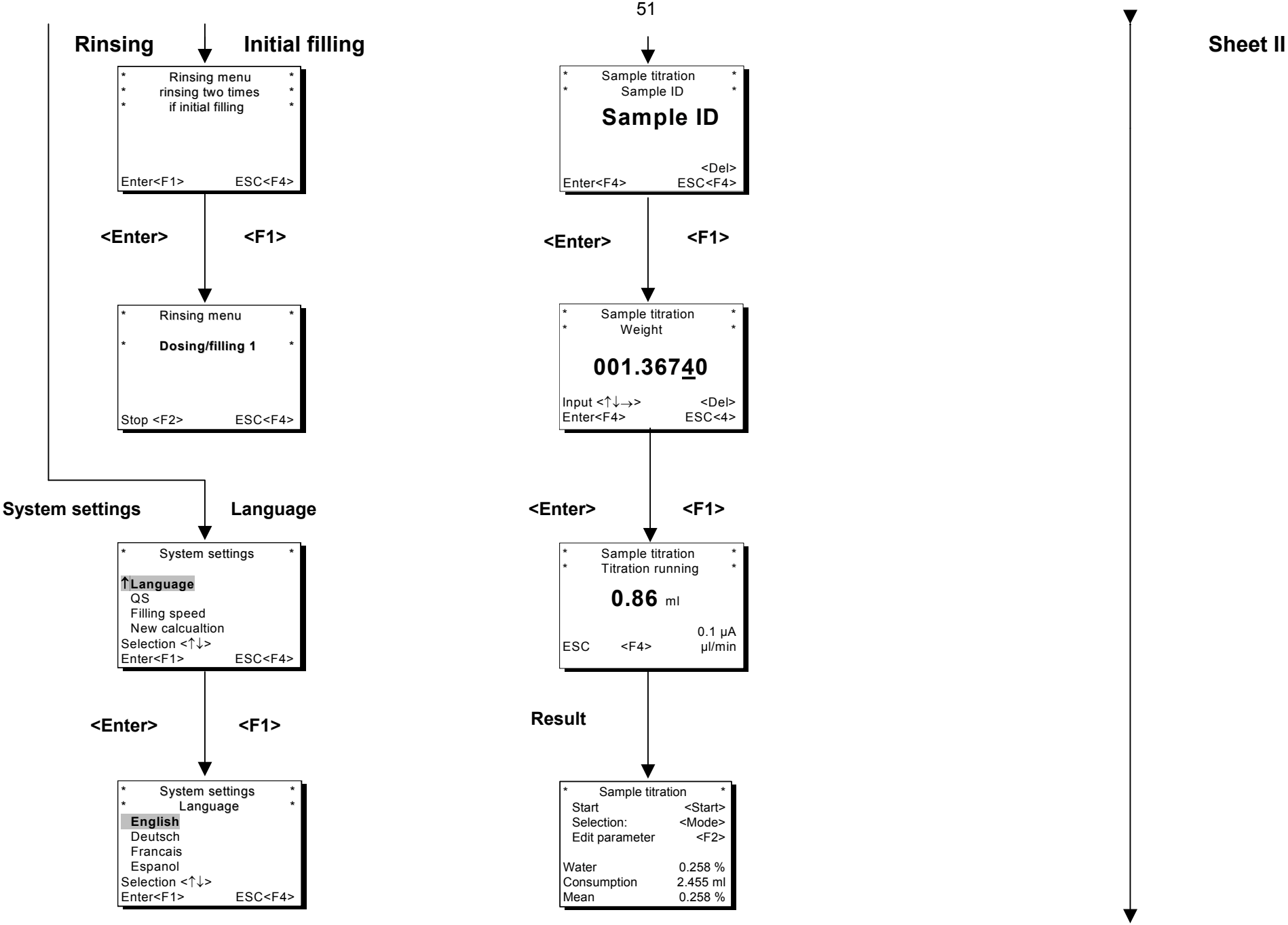
You can, of course, connect a bottle of a manufacturer of KF reagents equipped with a GL 45 thread directly to the adapter. Adapters for reagent bottles with an S 40 thread are also available (-> TZ 2008).

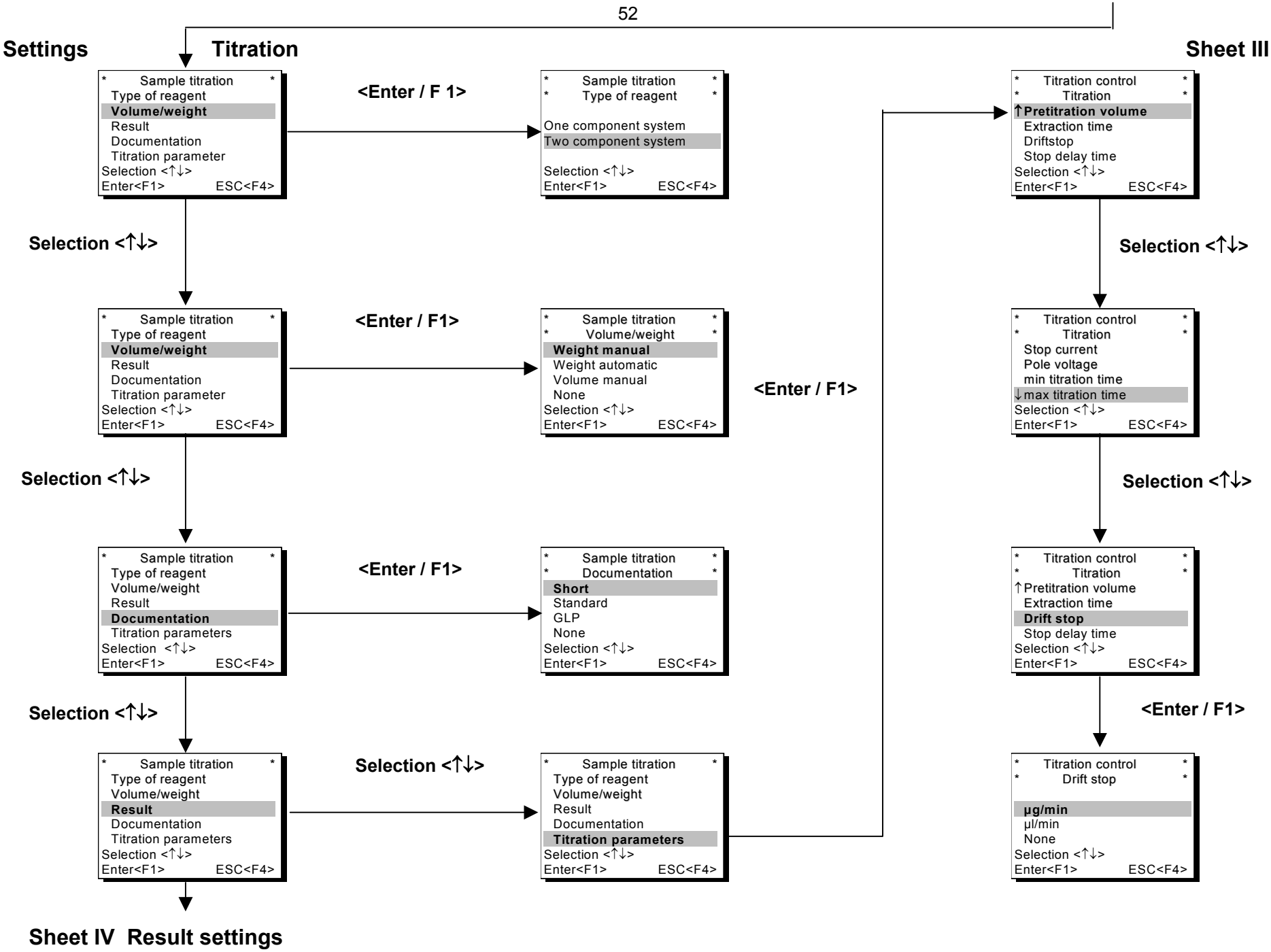
4 Working with the TitroLine *KF* Titrator

Sheet I

Menu structure

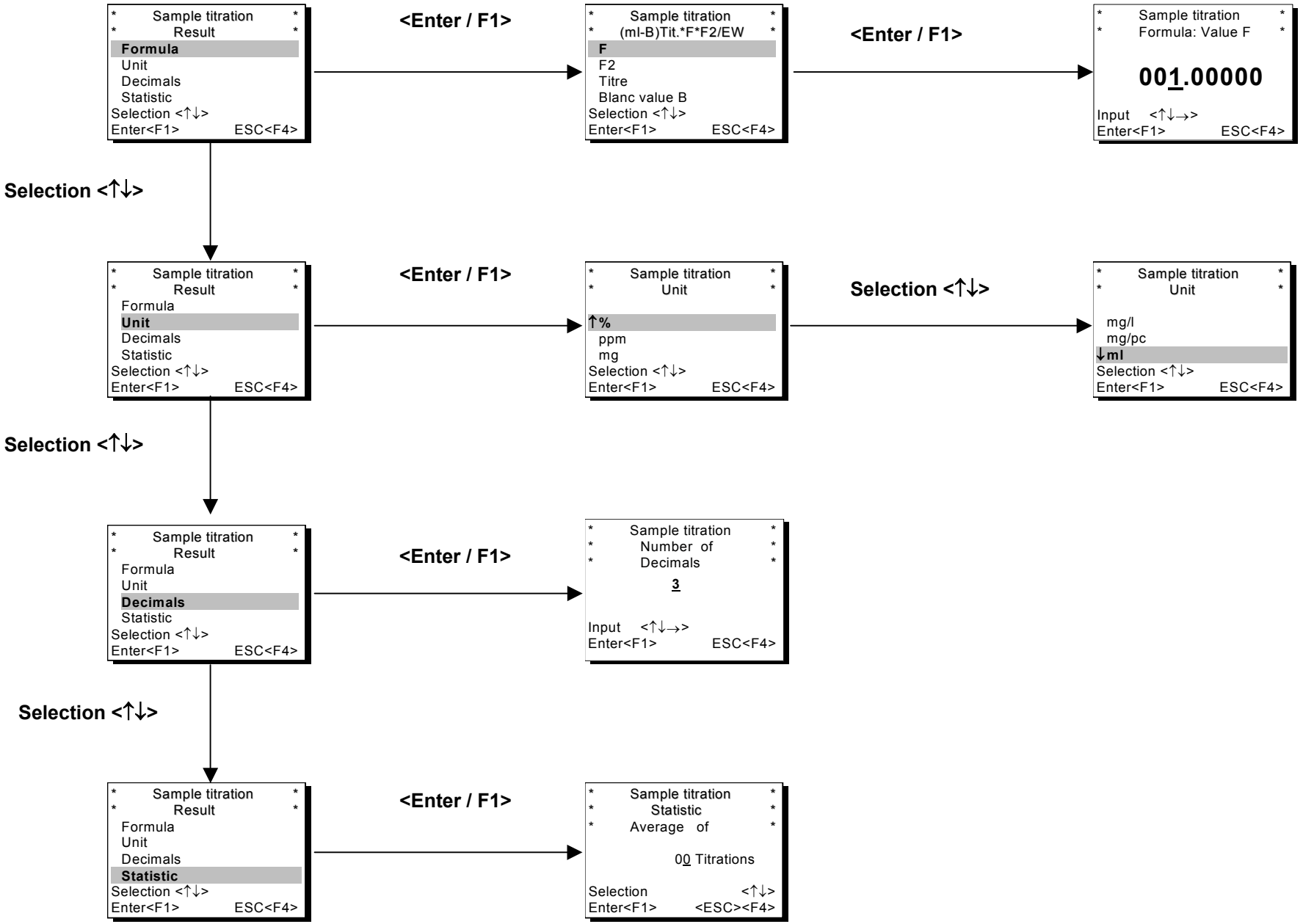



Rinsing
Initial filling
* Rinsing menu *
* rinsing two times *
* if initial filling *
Enter<F1> ESC<F4>
<Enter>
<F1>
* Rinsing menu *
* Dosing/filling 1 *
Stop <F2> ESC<F4>
System settings
Language
* System settings *
↑Language
QS
Filling speed
New calcualtion
Selection <↑↓>
Enter<F1> ESC<F4>
<Enter>
<F1>
* System settings *
* Language *
English
Deutsch
Francais
Espanol
Selection <↑↓>
Enter<F1> ESC<F4>
* Sample titration *
* Sample ID *
Sample ID
<Del>
Enter<F4> ESC<F4>
<Enter>
<F1>
* Sample titration *
* Weight *
001.36740
Input <↑↓→> <Del>
Enter<F4> ESC<4>
<Enter>
<F1>
* Sample titration *
* Titration running *
0.86 ml
0.1 µA
ESC <F4> µl/min
Result
* Sample titration *
Start <Start>
Selection: <Mode>
Edit parameter <F2>
Water 0.258 %
Consumption 2.455 ml
Mean 0.258 %
Sheet II


Settings
Titration
Sheet III
Sample titration
Type of reagent
Volume/weight
Result
Documentation
Titration parameter
Selection <↑↓>
Enter<F1> ESC<F4>
<Enter / F 1>
Sample titration
Type of reagent
One component system
Two component system
Selection <↑↓>
Enter<F1> ESC<F4>
Selection <↑↓>
Sample titration
Type of reagent
Volume/weight
Result
Documentation
Titration parameter
Selection <↑↓>
Enter<F1> ESC<F4>
<Enter / F1>
Sample titration
Volume/weight
Weight manual
Weight automatic
Volume manual
None
Selection <↑↓>
Enter<F1> ESC<F4>
Selection <↑↓>
Sample titration
Type of reagent
Volume/weight
Result
Documentation
Titration parameters
Selection <↑↓>
Enter<F1> ESC<F4>
<Enter / F1>
Sample titration
Documentation
Short
Standard
GLP
None
Selection <↑↓>
Enter<F1> ESC<F4>
Selection <↑↓>
Sample titration
Type of reagent
Volume/weight
Result
Documentation
Titration parameters
Selection <↑↓>
Enter<F1> ESC<F4>
Selection <↑↓>
Sample titration
Type of reagent
Volume/weight
Result
Documentation
Titration parameters
Selection <↑↓>
Enter<F1> ESC<F4>
<Enter / F1>
Titration control
Titration
↑Pretitration volume
Extraction time
Driftstop
Stop delay time
Selection <↑↓>
Enter<F1> ESC<F4>
Selection <↑↓>
Titration control
Titration
Stop current
Pole voltage
min titration time
↓max titration time
Selection <↑↓>
Enter<F1> ESC<F4>
Selection <↑↓>
Titration control
Titration
↑Pretitration volume
Extraction time
Drift stop
Stop delay time
Selection <↑↓>
Enter<F1> ESC<F4>
<Enter / F1>
Titration control
Drift stop
µg/min
µl/min
None
Selection <↑↓>
Enter<F1> ESC<F4>
Sheet IV Result settings

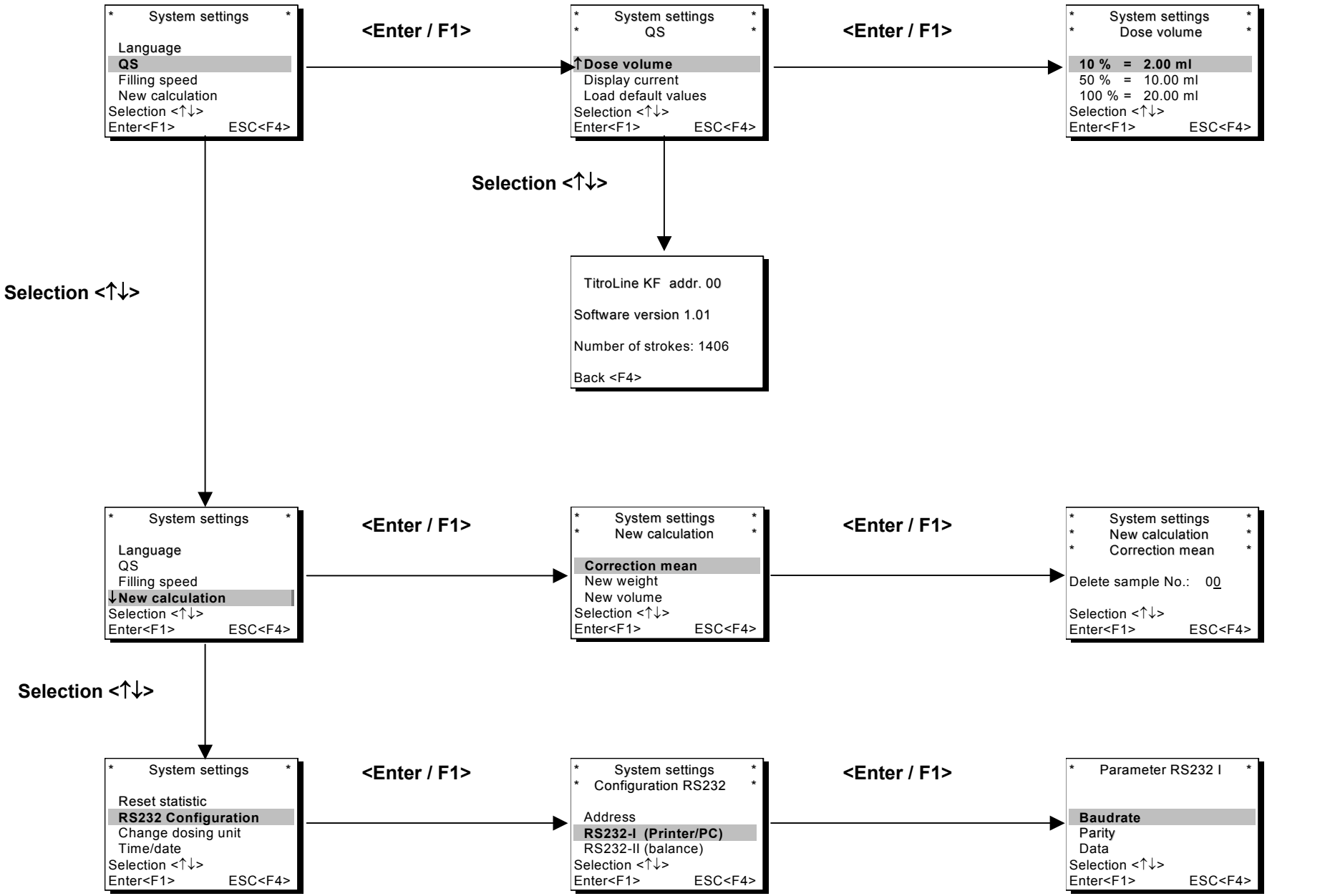
Result settings

Sheet IV



Further system settings

Sheet V



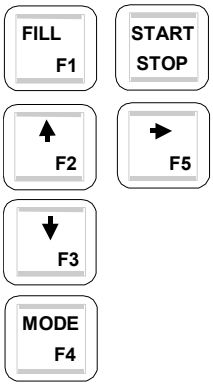
4.1 Keys and contrast

You can adjust the contrast of the display text using the knurled wheel on the right side of the titrator.

The TitroLine *KF* Titrator can be operated using the keypad on the front panel of the device **or** a connected PC mini keyboard.

TitroLine *KF* keyboard

Figure 6

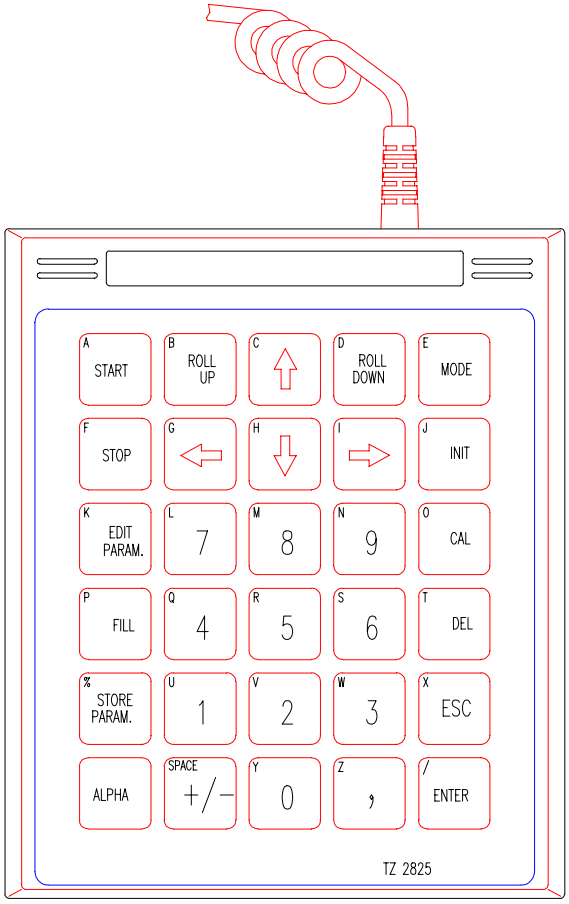


The device can be fully controlled using the keyboard on the TitroLine *KF* Titrator (🕮 **figure 6**). However, for the input of sample designations, and more especially for the input of weighing data, it is recommended to use the external PC mini keyboard (🕮 **figure 7**).

The keyboard on the device consists of six keys. The display on the TitroLine *KF* Titrator will show in each situation which key on the device itself or on the PC mini keyboard has to be depressed.

TZ 2825 PC mini keyboard

Figure 7



4.2 Switch-on image, main and selection menu

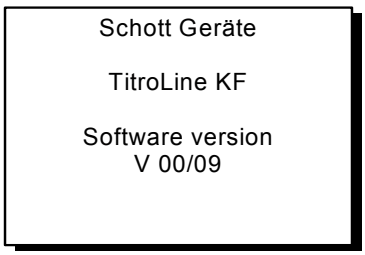


Fig. 8

After switching the TitroLine *KF* Titrator on, the **switch-on image** (🕮 **figure 8**) will show the name of the device and the software version. The switch-on image will be displayed for 15 s and then change automatically to the **main menu** (🕮 **figure 9**). The method last used will appear on the display.

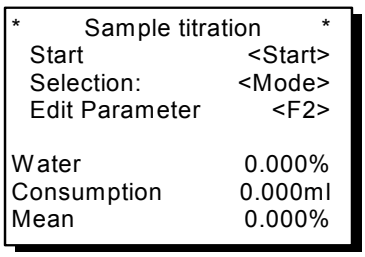


Fig. 9

From the main menu, use **<Start>** to start the current method. By selecting <**F2**> or <**Edit Param**.>, respectively, you will be taken to the parameter settings of the current method. <**Mode**> will take you to the **method selection menu** (🕮 **figure 10**).

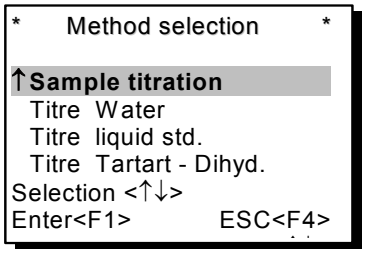


Fig. 10

Using the ↑ and ↓ keys, you select the desired method for the titer, blank value, or sample titration and confirm the selection using <**F1**> or the <**ENTER**> key. <**F4**> or <**ESC**> will take you back to the main menu.
Multiple depression of the arrow keys will take you to other selection options (🕮 **figure 11**).

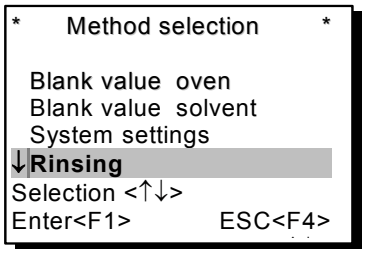


Fig. 11

Here you can also select the **Rinsing** rinsing program (initial filling) and the system settings.
The system settings include, for instance, also the setting of the national languages (🕮 **chapter 4.3**).

4.3 Selection of the national language

The TitroLine *KF* Titrator is ex-works set to English as national language. In order to select a different language (Deutsch, French or Spanish), use <**Mode**> or <**F4**> on the main menu (🕮 **figure 9**) to get to the method selection menu (🕮 **figure 10/11**) and from there to the system settings (🕮 **figure 12**).

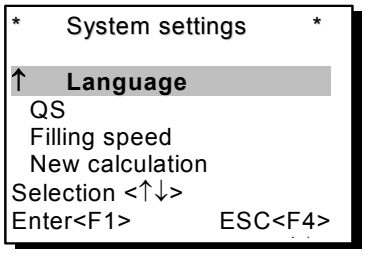


Fig. 12

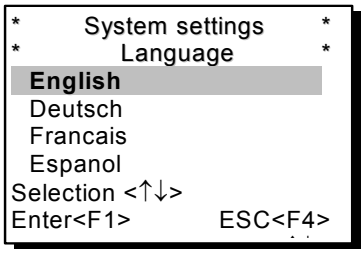


Fig. 13

The national language is selected using the **<↑↓>** arrow keys and confirmed using <**Enter**> or <**F1**>. The display will adjust to the national language selected and will jump automatically one level back to the system settings.

4.4 Rinsing and initial filling

If the reagent bottle is already filled with titration solution, or if the filled reagent bottle has already been connected, you can now fill the titrator with the KF titration solution.

⚠ *Please note* ⚠**:** The titration hose has to be properly connected to the valve and the titration vessel (🕮 **figure 1 or figure 55**)

⚠ *Please note* ⚠**:** The titration hose has to be properly connected to the valve and the titration vessel (🕮 **figure 1 or figure 55**)

Select **Rinsing** from the **method selection menu** (🕮 **figure 11**). Start at the rinsing operation with <**Enter**> or <**F1**> (🕮 **figure 14**).

Starting the function

Figure 14

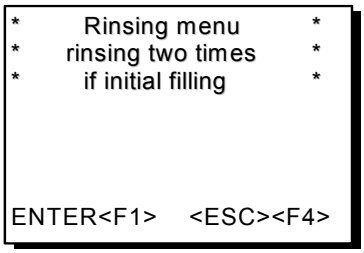
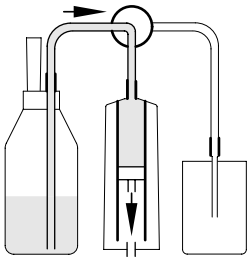
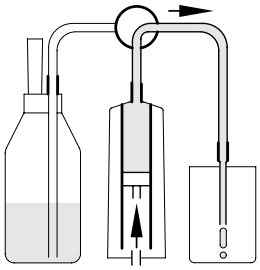


Figure 15





One drawing-in and dosing operation is triggered. Old solution which may be contained in the cylinder will be flushed out, subsequently a filling operation with the new liquid will be performed. Changing solutions in this way is not recommended, however, since the entire contents of the cylinder will not be ejected without residues, and thus a mixing with the new solution will take place.

The rinsing operation can be interrupted at any time using the <**STOP>** key, with rinsing being resumed by pressing the <**START>** key. If you press the <**Esc**>or <**F4**> key, the rinsing process is definitely cancelled, with the process being concluded by an automatic filling of the burette.

It should be noted that the titration solution can alter after just a few hours owing to diffusion of constituents of the air. In cases of doubt you should rinse as well.

4.5 Filling

The TitroLine *KF* Titrator fills the cylinder automatically after each titration. If the consumption of a titration exceeds 20 ml, an automatic filling processes will also take place. If the cylinder should not be filled for any reason, you may trigger a filling processes from the starting level / main menu using the <**FILL**> key.

4.6 Starting a titration

All preparatory work being done, you can now start with a titration or a titer determination (🕮 **chapter 4.7**). If not yet done, press the red key of the TitroLine *KF* Titrator to fill some solvent into the titration vessel. By the way: you will find further information and important tips on this topic in the enclosed "Karl-Fischer titration in practice" brochure.

Start the selected method with the <**Start**> key. The titrator will first start the conditioning process (🕮 **figure 16**). In this process the titration cell will be dry-titrated. The process will involve the entire water contents of the solvent and the leakage adhering inside the titration vessel.

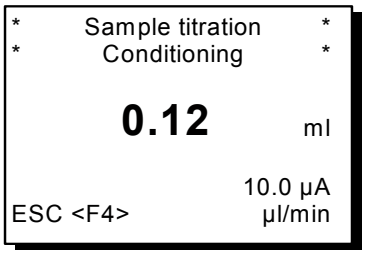


Fig. 16

During the conditioning process, the TitroLine *KF* Titrator indicates the titration-agent consumption in terms of ml, the current drift in µl/min, and the measurement value in µA. As soon as the titration cell is dry, the display will change as indicated in 🕮 **figure 17.**

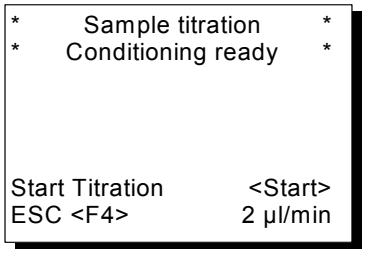


Fig. 17

Subsequently, another depression of the <**Start**> key allows you an immediate or delayed triggering of the actual sample titration, of a titer determination, or of a blank-value titration. The TitroLine *KF* Titrator will remain in a stand-by mode in which the titration cell is always kept dry, and thus ready for use. After pressing the <**Start**> key, (🕮 **figure 18**) will appear.

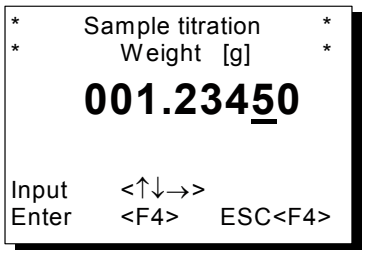


Fig. 18

The sample designations can only be input with a PC mini keyboard connected. Up to 16 alphanumeric characters can be input. <**Del**> will delete an input. The input is again confirmed with <**Enter**>. Without an external keyboard, the sample ID will automatically be counted upwards in increments of 1. You can now put the sample in the titration vessel.

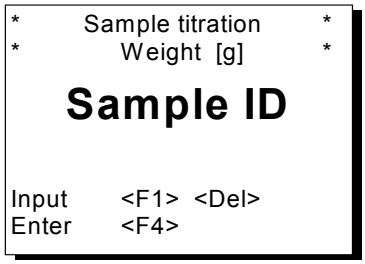


Fig. 19

At this point you can enter the pattern (weighed-in quantity in g or volume in ml) of the sample. Using the TZ 2828 keyboard, the numeric values are entered directly via the numeric keyboard. If no external keyboard is connected, use the **<→>**, **<↑>**, or **<↓>** arrow keys to set the numeric values. With the <→> arrow key, you select the digit to be set. You can change its value using the <↑> and <↓> arrow keys. Please confirm your input with <**Enter**> or <**F1**>. The titration will now be started with the preset parameters.

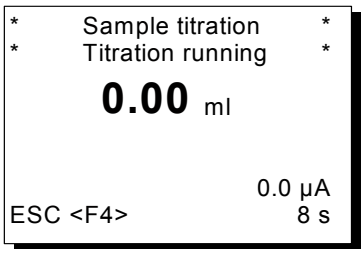


Fig. 20

First you have to wait till the extraction time has lapsed. An extraction time of 10 s is the set ex-works. The
extraction time and other titration parameters can be adjusted individually (🕮 **chapter 5.8**).
Following the extraction time, the actual titration will start. The titration will run until the pre-selected end criteria have been reached. As a rule, this is a specific drift and a specific current (🕮 **chapter 5.8**).

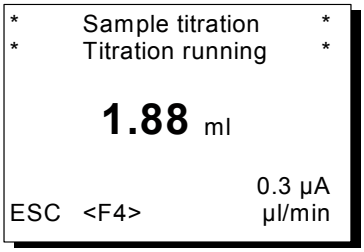


Fig. 21

In the course of the titration the display will show the ml consumption in large characters, whereas the drift of the titration cell and the current are indicated in small characters. The titration can be stopped at any time with the <**Esc**> key.

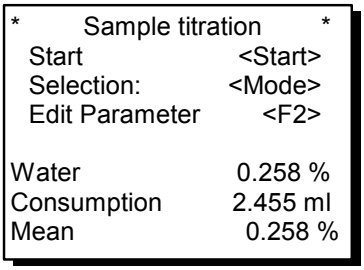


Fig. 22

At the end of the titration, the result is indicated on the display. The result will be present as water contents of the sample in % or in any other selected unit, the consumption in terms of ml, and the average value of the water contents. If a printer is connected, the result including other details will be printed on the printer.

Using the <**Start**> key you can start the next titration.

4.7 Titer determination

An accurate water determination requires an exact knowledge of the concentration of the titration solution. Although the reagent bottle shows already an approximate concentration of the solution (for instance, 5 mg/ml), the concentration of new solutions is as a rule higher and decreases over time. The enclosed certified standard solutions as well as the syringes with cannula allow a convenient determination of the titer.

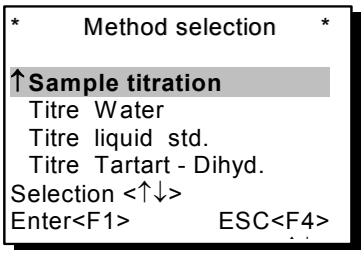


Fig. 23

From the main menu (🕮 **figure 22**) the <**Mode**> key will take you to the mode selection menu (🕮 **figure 23**). Select the "Titer liquid. Std." method using the arrow keys, then confirm your selection using <**Enter**> of <**F1**>. The display will automatically jump back to the main menu (🕮 **figure 24**). During first operation, "Titer liquid. Std." is preset, since a titer determination is required.

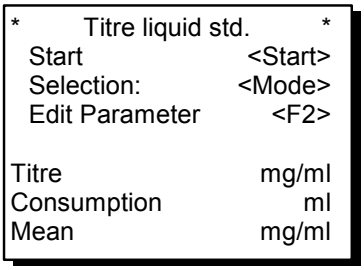


Fig. 24

At this point the desired titer method is loaded, and the titer determination can be started immediately.

The titer determination is ex-works set to a triple determination. This means, three titer determinations have to be performed, which are then automatically used to calculate a mean value which will be taken into account in sample titration.

- Take one ampoule from the box coming with the device. In this process please check the concentration of the standard which is indicated on the certificate. Within the method, the concentration is defaulted to 10.03 mg/g. If the standards show a different value (for instance, 10.00 mg/g), you can correct this value in the formula. The value can be found below the F factor. Please change this value using the menu overview (🕮 **chapter 4, sheet III and IV**).
- Open the glass ampoule at the marked predetermined breaking point, then draw approx. 2 - 2.5 ml (approx. 5 mg/ml in the case of titration solution) or 0.75 – 1 ml (approx. 2 mg/ml in the case of titration solution) in to a syringe with needle. Start the method so that the TitroLine *KF* Titrator can condition the titration vessel.
- Place the syringe in a beaker glass on a weighing-balance, and press the tara-weight key (zero key).
- As soon as the titrator has completed conditioning, press the <**Start**> key. The titrator will ask you for the weighing-in value of the standard.
- Inject the standard solution through a septum into the titration vessel.
- Put the syringe back on to the weighing-balance, then press the print key on the weighing-balance, or read off the value in terms of grams without sign (absolute value).
- If the weighing-balance is directly connected to the titrator, titration will start automatically. Otherwise you input the weighed-in quantity via the keyboard in the TitroLine *KF* Titrator, then confirm the value. The titrator will then also start titration.
- Repeat this process 3 times or as many times as you have preset within the method.

For further details on titer determination and handling, please refer to the "Karl-Fischer titration in practice" brochure.

5 Methods parameter

5.1 General

The methods in the TitroLine *KF* Titrator are ex-works defaulted to customary parameters. However, the user can adapt the parameters of the individual methods to his own requirements. <EDIT Parameters> or <F2> will take you from the main menu to the method parameters (🕮 **figure 25**).

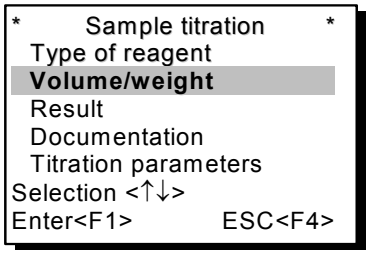


Fig. 25

For type of reagent you can choose between a one component or a two coponent system. Please refer to the “Karl-Fischer titration in practice” brochure.

Under **Volume/Weighed-in quantity** you can select whether you wish to input a pattern in terms of ml or a weighed-in quantity in g, and whether the weighed-in quantity is to be entered manually or automatically.

Under **Result** you can modify the factors of the formula, select the units, set the decimal digits, and activate statistics.

Under **Documentation** you can select the form of the documentation among: “short”, “Standard”, “GLP”, “Display only”.

The actual parameters of the titration are to be set under "**Titration parameters**". The following parameters can be set for the titration:

- the pretitration volume [ml]
- the extraction time [s]
- the drift stop [µl/min] or [µg/min]
- the switch-off time [s]
- the switch-off current [µA]
- the pole voltage [mV]
- min. and max. titration duration [s]

5.2 Volume and weighed-in quantity

Manual weighed-in quantity is set for “**Sample titration**”, “**Titer water**”, “**Titer liquid standard**”, and “**Titer tartrate-dihydrate**” methods (🕮 **figure 26**). So if somebody has connected a weighing-balance to the TitroLine *KF* Titrator, it makes sense to switch to “**Automatic weighing-in**”. In this case it takes just a keystroke on the weighing-balance to send the weighed-in quantity directly to the titrator. The weighed-in quantity will appear on the display where it is confirmed with <ENTER> or <F1>.

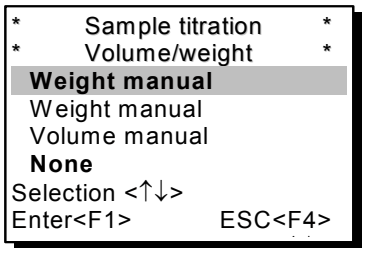


Fig. 26

If weighed-in quantities or patterns are to be transferred directly to a connected computer, you should select "None".

In the “**Blank value oven**” and “**Blank value solvent**” the “**Volume/Weighed-in quantity**” method parameter is not active.

Volume manual is only used for the "**Titer water**" method, if a highly accurate microlitre syringe is used for the addition of the water.

5.3 Result – formula

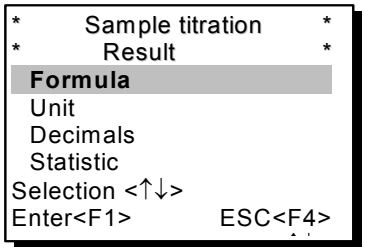


Fig. 27

Via method parameters (🕮 **figure 25**) you get to the **Results menu**. With <**ENTER**> you get to the formula editor (🕮 **figure 28**).

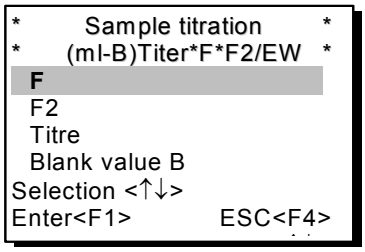


Fig. 28

Three different formulae are used in the TitroLine *KF* Titrator. The formula shown in 🕮 **figure 28** is only used for the "**Sample titration**" method.

The following formula is used for the various methods of titer determination:

EW*F*F2/((ml-B)*F3)

The following formula is used for the two blank-value methods:

ml*F*F2*F3

Legend:

ml	consumption of KF reagent
F	conversion factor, default value = 1.000
F2	conversion factor, default value = 1.000
F3	conversion factor, default value = 1.000
Titer	concentration of the KF reagent in terms of mg/ml
Blindwert B	default value = 0.000
EW	weighed-in quantity in g or volume in ml, default value =1.000

In the case of sample titration, an internal conversion factor is calculated for the selected units % (standard) and ppm. In the case of the % unit, the conversion factor is 0.1, for ppm the conversion factor is 1000. With these factors the result is calculated correctly right from the onset, and as a rule no further input is required.

The titer methods **always** calculate the result in terms of mg/ml. No selection of the unit has to be done here. In the case of the "Titer water" method, an internal conversion factor of 1000 is also calculated. With the "titer liquid standard" method, **F** is defaulted to the numeric value of 10.03. The numerical value of 10.03 corresponds to the concentration of the enclosed liquid standards. If the standard has a different concentration, the F value has to be modified correspondingly. In the case of the "Titer tartrate dihydrate" method, F is defaulted to 156.6. This value is derived from the water contents of the standard for the sodium-tartrate-2-hydrate KF titration. These values can be changed if required.

Input of numeric values

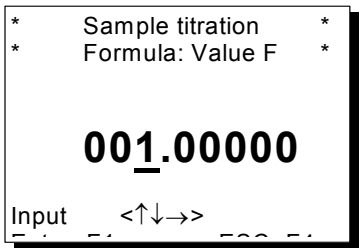


Fig. 29

Input using with the keypad on the TitroLine *KF* Titrator:

The desired digit is selected using the → key. The numeric value can be changed with ↑ and ↓. The input is confirmed with the <**F1**>.

Input using connected PC mini keyboard:

Simply enter the numeric value using the numeric keys. Using <**Del**>, an individual numeric value can be deleted again. The input is confirmed using <**ENTER**>.

5.4 Result – unit

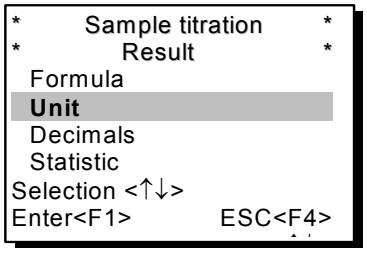


Fig. 30

From the method parameters (🕮 **figure 25**) you get to the results menu (🕮 **figure 30**). Using <↓> and <**ENTER**> or <**F1**> you get to the units sub-menu (🕮 **figure 31**).

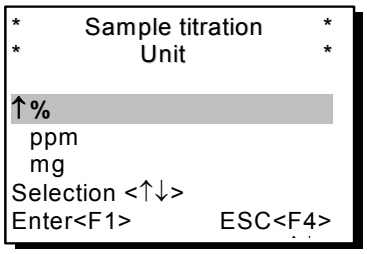


Fig. 31

The **%, ppm, mg**
(🕮 **figure 31**)

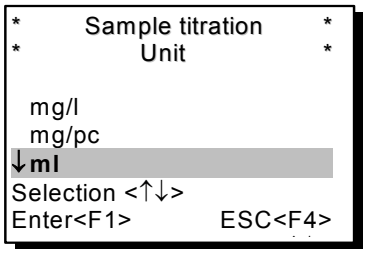


Fig. 32

mg/l, mg/pc (pc = piece) and
ml units are available for selection
(🕮 **figure 32**).

5.5 Result – decimals

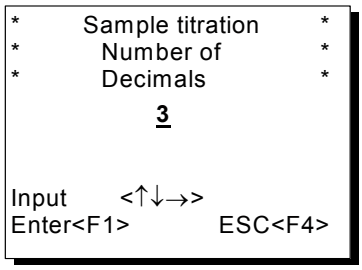


Fig. 33

Starting from the results menu (🕮 **figure 30**), you get to the decimal digits (🕮 **figure 33**). The decimal digit of a result can here be changed using <↑> or <↓> or by the direct input of the numeric value. The default value for all methods is **3**. Up to five decimal digits can be set. The input is confirmed using <**F1**> or <**ENTER**>.

5.6 Result – statistics

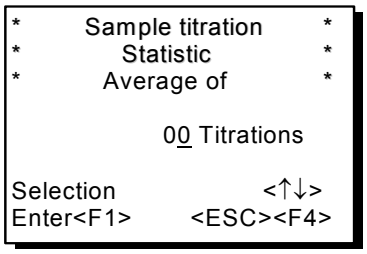


Fig. 34

Starting from the result menu (🕮 **figure 30**) you get to statistics (🕮 **figure 34**). If you want to calculate an average value for a sample out of a multiple determination, this is the point for inputting the number of single measurements. In the case of a double determination, for instance, the numeric value of 2 would have to be entered here. The default value for both sample titration and blank-value methods is 0. For titer determinations the default value is 3. In addition, if a printer is connected, the standard deviation and the variation coefficient (relative standard deviation) will be output as well.

5.7 Documentation

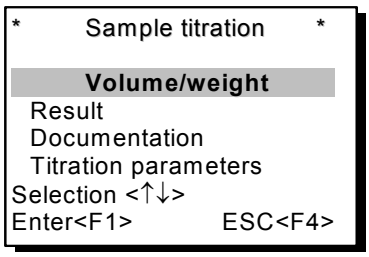


Fig. 35

<Edit PARAMETER> or <**F2**> will take you from the main menu to the method parameters (🕮 **figure 35**). Pressing <↓> twice, followed by <ENTER> or <**F1**>, you get directly to the documentation menu (🕮 **figure 36**).

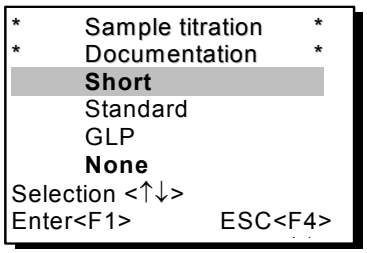


Fig. 36

At that point he can select out of four different forms of documentation. The presetting is **short**. It is used if no printer is connected. The display will always show the water contents, the consumption, and the mean value of the water contents. This applies to the other documentation forms as well. **Table 1** shows the differences between the various forms of documentation.

Tab. 1

	Documentation forms		
Documentation parameters	**Short**	**Standard**	**GLP**
Consumption	**X**	**X**	**X**
Water contents	**X**	**X**	**X**
Statistics: mean value, Std, relartive Std.	**X**	**X**	**X**
Weighed-in quantity/Pattern: g or ml	**X**	**X**	**X**
Date: 31.03.2000		**X**	**X**
Time: 14.23.59		**X**	**X**
ID: alphanumeric	**X**	**X**	**X**
Titer		**X**	**X**
Blank value		**X**	**X**
Drift		**X**	**X**
Titration duration		**X**	**X**
Method		**X**	**X**
Titration parameters			**X**
Calculation formula + values			**X**
User input field			**X**

5.8 Titration parameters

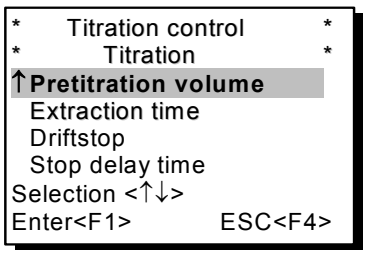


Fig. 37

From the method parameters, pressing <↓> and <ENTER> three times or <F1> will take you directly to the titration parameters (🕮 **figure 37**). The term of titration parameters refers to those parameters controlling the speed and the course of the titration. One may therefore also speaks of controlling parameters.

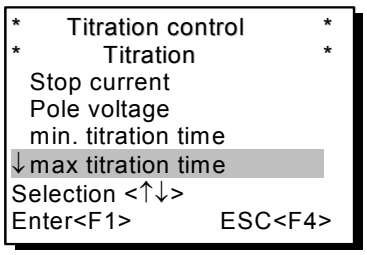


Fig. 38

Pressing <↑> once or <↓>four times will take you to the further selection of controlling parameters (🕮 **figure 38**).

Pretitration volume

The pretitration volume will dose in one step after the start of the titration a certain volume into the titration vessel. The pretitration volume can reduce the titration time when having a very high water content. Volume settings are possible between 0.1 and 99.9 ml. The standard value of 0.0 ml is set for all methods.

Extraction time

After the addition of the sample into the titration vessel you have to wait for a certain period of time before the TitroLine *KF* Titrator will start with the addition of the KF reagent. This waiting time is necessary either to dissolve the sample or to extract the water from insoluble sample. This is why this time is called extraction time. The extraction time can be set between 0 and 9999 s. In the case of the "Titer sodium tartrate-dihydrate", the extraction time is preset to 145 s, with all other methods the preset team is 10 s.

Drift stop

Some humidity from the environment will penetrate into each and any titration vessel, even if it appears to be very tightly closed. As a matter of course, this humidity consumes KF reagent. So the term of drift refers to the consumption of the KF reagent over time. The drift is displayed automatically during conditioning and in the course of titration. The drift is indicated in µl/min, in the case of a known contents of the KF titrant, in µg/min. This drift can also be used as a very useful cancellation criterion. The value can be entered from 2 bis 100 µg/min.

Switch-off time

If the set switch-off current reaches the switch-of time entered here, the titration will be cancelled. All methods are preset to a standard value of 10 s.

Switch-off current

The switch-off current in terms of µA can be set variably between 1 and 100 µA. Its standard presetting for all methods is 20 µA, and as a rule it has not to be changed.

Pole voltage

The pole voltage is the voltage which is applied between the two platinum pins of the double platinum electrode. The current values measured will vary as a function of the voltage being applied. The standard presetting for all methods is 100 mV. The value can be set from 10 - 200 mV. If you wish to use different solvents at a later time, you can optimise the titrating speed further by modifying the pole voltage.

min. titration duration

The minimum titration duration is a suitable parameter to be used for samples with which the water can be extracted only slowly. In the case of such samples it makes sense to add KF reagent in order to have the water react away as early as during the extraction. In this way the water is extracted more quickly from the sample. The minimum titration duration can be set between 0 and 9999 s. The standard value for all methods is preset to 0 s. This means it is not activated.

max. titration duration

The maximum titration duration is used in the case of samples which create an increased drift towards the end and in which no end point stable curse, for instance, as a result of a slow extraction of water. The minimum titration duration can be set between 0 and 9999. The standard value for all methods is preset to 0 s. This means it is not activated.

6 System settings

The setting of the national languages is already explained in 🕮 **chapter 4.3**.

6.1 QS

QA = Quality Assurance. Quality assurance has reached an outstanding importance over the past years, in particular in sensitive ranges of the pharmaceutical and food industries. One major issue of quality assurance is the supervision of measurement apparatus. In the case of titration, this is as a rule done in the form of a gravimetric examination of the dosed volume according to DIN 12650, Part 6, or DIN EN ISO 8655. The procedure consists in a multiple dosing of 10, 50, and 100 % of the burette volume, subsequently these dosing volumes are to be weighed on an analytical weighing balance. The “QA - Dose volume” menu will support you in this procedure.

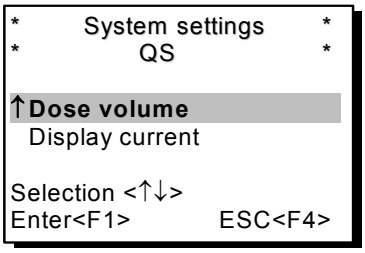


Fig. 39

Via the “Method selection menu” and “System settings” you get to the QA menu (🕮 **figure 39**). The QA menu contains three functions. The “Display measurement value” function can be used to check the measurement-value display and the measurement input. <**ENTER**> or <**F1**> will take you to “Dose volume" (🕮 **figure 40**).

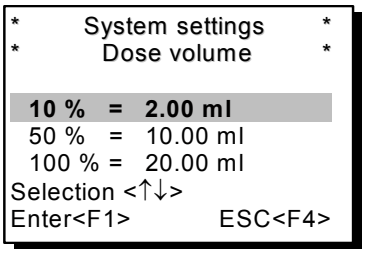


Fig. 40

The required volumes are already preset. Select the desired dosing volume and start dosing with <**ENTER**> or <**F1**>. For further information on the verification according to DIN 12650, Part 6, or DIN EN ISO 8655, please refer to "Karl-Fischer titration in practice" brochure.

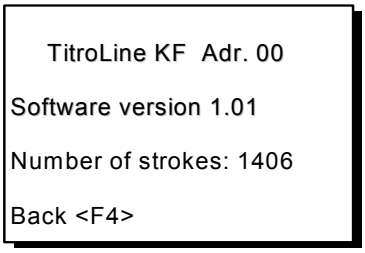


Fig. 41

<↑↓> will take you from the “Dose volume” or “Display measurement value” function to an informative display. This informative display contains the name of the device, the software address, the software version, and the total number of strokes or partial strokes performed by the titrator.

6.2 Filling speed

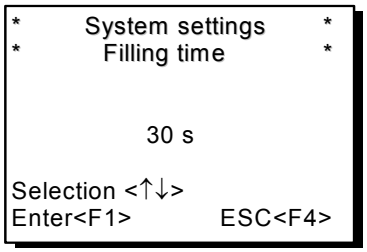


Fig. 42

The system settings also contained the setting of the filling speed. Properly speaking, it is not the speed which is set, but rather that time in terms of s which the TitroLine *KF* Titrator requires for one filling operation. The value of 30 s is the smallest possible value and at the same time the standard value. If a titration solution has the tendency of exhalation or a higher viscosity than those handled before, this time can be increased to, for instance, 60 s.

6.3 New calculation

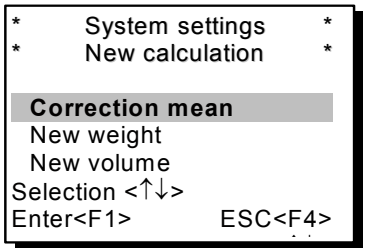


Fig. 43

Using “Re-calculation” (🕮 **figure 43**), the customer has the possibility of correcting a calculated result. A result correction may be required, for instance, if the weighed-in quantity of the pattern were input incorrectly. A correction of the mean value resulting from a multiple determination is also possible by deleting a "maverick" (freak value).

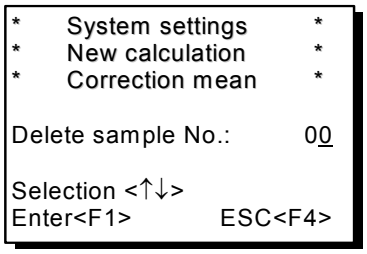


Fig. 44

Deleting a single result from a multiple determination should be considered very carefully. In cases of doubt, you should rather perform a new test series. For instance, if you performed a quadruple determination prior to a titer determination, and if the first value is significantly different from the three other values, you would have to delete the first value 01. A new mean value will be calculated automatically and displayed on the display and/or printed.

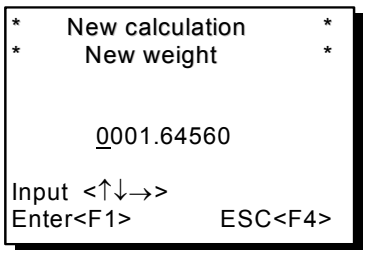


Fig. 45

If the weighed-in quantity or pattern of the **last** titration was input incorrectly, you can input the correct value of the weighed-in quantity of pattern at this point. The display will show the current weighed-in quantity/pattern. If this value was corrected at this point, please confirm your input by <**ENTER**> pr <**F1**>. A new result will be calculated automatically and displayed on the display and/or printed.

All of the newly calculated values will be indicated with an identifying mark.

***** A significant deviation is to be assumed, as a rule, if the value deviates by more than four standard deviations from the mean value of the other values. The standard deviation is also calculated on the basis of the other values.

6.4 Reset statistics

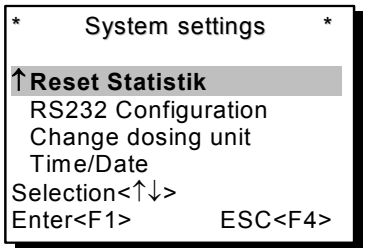


Fig. 46

If the status of the mean value in the case of a multiple determination should be unclear, you can use this menu (🕮 **figure 46**) to delete the statistics memory. Only the statistics memory of the currently selected method, for instance, "Titer determination water", will be deleted. To be sure, you will be prompted for 'yes' on 'no' after invoking this function.

6.5 RS-232 configuration

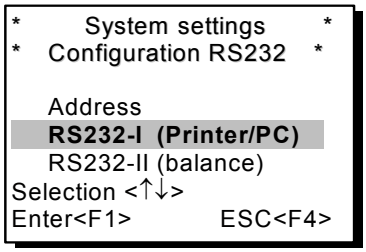


Fig. 47

The two RS-232 interfaces of the TitroLine *KF* Titrator can be configured in different ways, if, e.g. with a connected weighing-balance, a different configuration is to be set than with a printer being connected simultaneously. The two RS-232 interfaces are ex-works set to the following parameters

Baud rate: **4800**, parity: **no**, data: **7**.

The device address is preset to 0. It can be configured up to address 15. It may be changed, for instance, if multiples devices (TitroLine *KF* Titrator, TITRONIC® *universal* Piston Burette etc.) are linked together in one chain, also refer to 🕮 **chapter 7.2**.

6.6 Replacing the dosing unit

If it should happen that the dosing unit needs to be replaced, please note the following warning information:

⚠ **Caution! Please wear protective glasses!** ⚠

The dosing unit is equipped with lateral ribs arranged at 6 points around its circumference, with one of these ribs being in double design (**figure 48**, item 2). This double rib serves as a mark for the correct placement of the dosing unit.

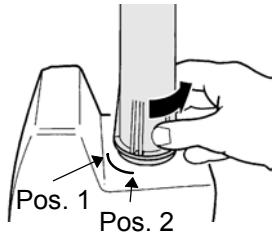


Fig. 48

After selecting and confirming "Replace unit" in the "System settings", you can unlatch the unit. To do so, please proceed as shown in (🕮 **figure 48**), i.e. by a slight rotation to the left, i.e. anti-clockwise. Subsequently, please press <**ENTER**> or <**F1**>. The piston will move upwards.

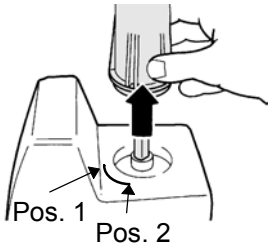


Fig. 49

After the piston has moved upwards by approx. 80 %, you are requested to unlatch the unit. If this has already been done at the beginning, please proceed with <**ENTER**> or <**F1**>. If the piston has moved out completely, you can remove the unit (🕮 **figure 49**).The display will show <Remove unit/Put on new unit>.

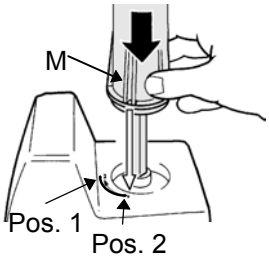


Fig. 50

Please align the new dosing unit according to the indication mark (double rib!), place it on perpendicularly (🕮 **figure 50**), then press <**ENTER**> or <**F1**>. The motor will now pull the dosing unit downwards, while the display requests you to align the unit. If you should hear any noise (rattling) at this point, please interrupt this process immediately using the <Esc> key. The cause of this "rattling" noise will be eliminated by setting on the dosing unit once again perpendicularly.

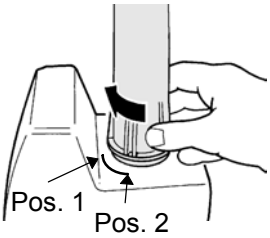


Fig. 51

The display will now ask you to fasten the dosing unit by rotating it one quarter to the right (clockwise) (🕮 **figure 51**). To conclude, please fasten the hose to the glass cylinder again.

6.7 Replacing the titration solution

If the unit is not replaced as a whole, a substitution of the titration solution by another one generally involves certain mixing and carry-over processes. The reason for this is to be found in the dead volume above the piston in the cylinder and in the hoses. The disturbances to be anticipated are the greater, the more the new solution differs from the previous type and concentration.

If the decision to change the titration solution has been made, the first thing to do is to remove the dosing unit as is described in 🕮 **chapter 6.6**). If possible, the residue of the titration solution should be removed manually by pushing the projecting piston rod carefully towards the hose connector. When doing so, more liquid will leak out of the burette tip, and the KF reagent is removed almost completely. Subsequently, the dosing unit is set on again (🕮 **figure 50**).

6.8 Date and time

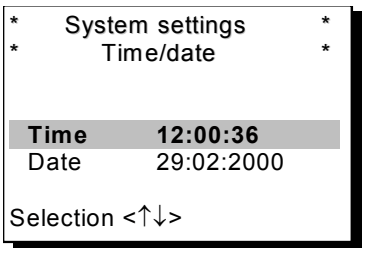


Fig. 52

The current time and date will be displayed if you select "Time/Date" in the system settings (🕮 **figure 52**). If the time or the date need to be corrected, please select the corresponding menu item and confirm your input with <**ENTER**> or <**F1**>.

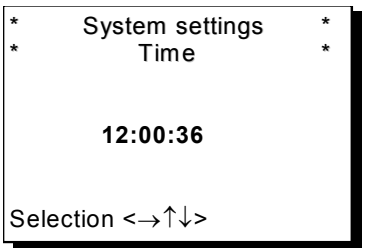


Fig. 53

The time is displayed in the hh:mm:ss format.

Foil keyboard: Use the → key to select the value to be modified, then change it using the ↑↓ keys. Conclude your input with the <**F1**> key.

External keyboard: Enter the entire time digit by digit, including the colons.

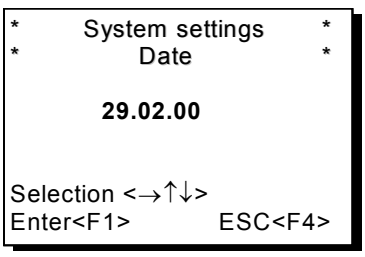


Fig. 54

The date is displayed in the dd:mm:yy format.

Foil keyboard: Use the → key to select the value to be modified, then a change them using the ↑↓ keys.

External keyboard: Enter the entire time digit by digit, including the dots. Conclude your input with the <**ENTER**> key.

7 Data communication via the RS-232 interfaces

7.1 General

The TitroLine *KF* Titrator is equipped with two serial RS-232-C interfaces for data communication with other devices. You can use these two interfaces to connect several devices to one computer (PC) interface.

RS-232-C socket "1" is used to establish the connection to a connected computer, to a printer with a serial interface, or to the preceding device in the "daisy chain". RS-232-C socket "2" can be used to connect additional devices (Daisy chaining concept).

PIN assignment of the RS-232-C interfaces:

PIN no.	Signifiacation / Description
1	T x D data output
2	R x D dat input
3	Digital mass

7.2 Chaining of several TitroLine *KF* Titrators — Daisy Chain concept —

In order to enable you to individually address several devices in a chain, each of the devices must have an address of its own. To achieve this, one has first of all to use an RS-232-C data cable, e.g. no. TZ 3098 type, to establish a connection between the computer and the RS-232-C interface 1 of the first device in the chain. Using another RS-232-C data cable, no. TZ 3094 type, the RS-232-C interface 2 of the first device is connected to interface 1 of the second device. It is possible to connect another device to interface 2 of the second device.

The address always consists of 2 characters: e.g. address 1 of the two <0> and <1> ASCII characters. The address can be set from **00** to **15**, i.e. there are 16 possibilities. Please ensure that the addresses of the devices in the chain are different. If a device is addressed using its address, the device will work off this command without sending it to another device. The response to the computer also receives the own address. The addresses are set as described in 🕮 **Chapter 6.5**.

The TitroLine *KF* Titrator will receive commands from a computer at interface **1**, if the latter carries this address, and this interface will also be used to return its response. If the address of the incoming commands does not match their device address, the entire command will be forwarded at interface **2**. This interface 2 is connected to interface 1 of another titrator/burette. This device, in turn, will now check the address and will respond to this command just like the first TitroLine *KF* Titrator. Any information (data strings) arriving at the interface 2 of the TitroLine *KF* Titrator will be output at interface 1 to the computer immediately. This means that the computer will in any case receive the information of all burettes. In practice it is possible to connect up to 16 devices to a computer (PC) interface.

7.3 Command list for RS communication

The commands are made up of the following 3 elements:	address	2-digit aa,	e.g..: 01
	command		e.g..: BP
	variable, if required		e.g.: 14
	and end of command		<CR> <LF>

Each command has to end on the <CR> and <LF> (Carriage Return and Linefeed) ASCII characters. All responses will only be returned to the computer upon completion of the respective action.

Example: It is intended to send to the titrator with the address 5 the command to dose 12.5 ml, with preceding filling of the cylinder and zero reset of the display.

The command consists of the following characters: 05DO12.5<CR LF>

Here is:	05	=	device address
	DO	=	command for dosing with filling
	12.5	=	volume to be dosed in ml
	<CR LF>	=	control character for end of volume

All TitroLine *KF* Titrators will, irrespective of their address, respond to address 99. The 99AA1 command automatically attributes to the individual devices the address from 01 to nn. So it is e.g. possible that addresses starting from 05 are output. In this case all devices will then respond with their new address. If a command is preceded by AB, all the TitroLine *KF* Titrators will work off the command and report back automatically. This means that one command can fill all burettes. The corresponding command is: 01ABBF<CR LF>.

Command	Description	Answer
99AAnn	Transmit new address (nn = new address)	nnY
aaAB*command*	Command after instruction AB ... is performed and sent to following device	aaY
aaBF	Fill piston burette	aaY
aaBN	Reset ml display to zero	aaY
aaBP	Inquiry of piston position in scale parts	aaBP=0 - 8000
aaBV	Inquiry of burette volume	aaV=12.34
aaDA13.56	Dose with addition of volume	aaY
aaDO123.4	Dose indicated quantity in ml	aaY
aaDB123.4	Dose quantity in ml without filling and zero	aaY
aaDH	Endless dosing, to be stopped	
aaDZ	Start dosing, without filling; display will add up to max. volume 9999 ml	aaY
aaGDM20,3	Dosing rate in ml/min	aaY
aaGF30	Filling time in s	aaY
aaKA	Which key is being pressed?	AaKey:*"Text"*
aaKD	Keyboard disable	aaY
aaKE	Keyboard enable	aaY
aaRA	Interchangeable unit - inquiry of volume size	aaUNIT:20
aaRH	Identification request	aaIdent:TL *KF*
aaRC	Send last command	aa"last command"
aaRS	Report status	aaStatus:*"text*
aaSC	Continue after stop / continue function after stop	aaY
aaSH	Stop, burette stops	aaY
aaSD1..8	Write string in n^{th} matrix line: attach string to be written with "TEXT" (limit characters to 21) as a delimiter character to command e.g.: 01SD11" ("Please replace interchangeable unit")	aaY
aaST0	Delete display completely	aaY
aaST1	Switch display fully on	aaY
aaSR	Stop current function	aaY
aaSYS5	Set display language to German	aaY
aaSYS1	Set display language to English -english-	aaY
aaSYS2	Set display language to French -français-	aaY
aaSYS3	Set display language to Spanish -español-	aaY
aaVE	Software version number	aaVersion:99.23
aaVO	Volume inquiry from display	aaV=12.345ml

8 Connecting analytical balances and printer

8.1 Connecting analytical balances

Since, as a rule, the sample or the standard will be weighed-in in on an analytical weighing-balance, it makes also a sense to connect this balance to the TitroLine *KF* Titrator. To be able to connect the balance to one of the TitroLine *KF* Titrators, the balance has to be equipped with an RS-232-C interface, and a suitably configured connection cable has to be available. Ready-made connection cables are available for the following balance types:

Balance	**TZ number**
Sartorius (all types)	TZ 3092
Mettler AT, PR, PM	TZ 3093
Mettler, AB-S, AG, PG	TZ 3099

Upon request, ready-made connection cables are available for other balance types. For these cables, however, we need detailed information about the RS-232-C interface of the balance being used.

The connection cable is connected to the RS-232-C interface of the TitroLine *KF* Titrator. This end of the collection cable is always made up of a 4-channel mini plug. The other end of the cable, depending on the balance type, consists of a 25-channel plug (Sartorius), a 9-channel plug (Mettler ABS), or a 15-channel special plug (Mettler AT).

In order to send the balance data to the titrator, the data transfer parameters of the titrator and the balance have to match. In addition, some basic settings have to be performed on the balances:

- The balance is supposed to send only after a print command
- The balance should send the weighing-balance data only upon the standstill of the display
- The balance should never be set to “send continuous”, “automatic spending”, or “continuous sending
- “Handshake” on the weighing-balance has to be set to “off”, “software handshake”, or “pause”
- No special characters such as **S** or **St** must be trailing in the data string of the balance data. This might prevent the proper processing of the balance data by the TitroLine *KF* Titrator.

After connecting the balance to the TitroLine *KF* Titrator using the proper cable, and after making the settings in the software of the balance including any adaptations to the TitroLine *KF* Titrator, if applicable, the transmission of the balance data can be checked very easily. Start the sample titration. After conditioning, start the titration using <Start>.
The display will request you to:

a) Press the print key on the balance → Parameter to "automatic weighing-in"

b) Enter the weighed-in quantity → Parameter to "manual weighing-in"

Place an object on the balance, then press the print key. After the standstill of the display on the balance, you will hear a beeping sound on the TitroLine *KF* Titrator, and the balance data transfer will appear as follows:

a) shortly in the display, and the display on the TitroLine *KF* Titrator will automatically change to the input of the sample designation

b) the weighed-in quantity has to be confirmed once again with <**ENTER**> or <**F1**>.

c)

8.2 Printer connection

The serial interface 1 of the TitroLine *KF* Titrator can be used to connect printers with an RS-232-C interface. The connection requires a TZ 3095 data cable (25-channel interface). In order to enable the results and further data to be sent to the printer, the data transfer parameters of the titrator have to match those of the printer. The transfer parameters of the TZ 3460 printer from Schott are already properly configured, and the TZ 3090 data cable comes with the scope of delivery of the TZ 3460 printer.

9 Trouble shooting

The device does not respond to keys being pressed, the display is dark.

Problem	Solution
The internal program is disturbed (e.g. by electrostatic charging or mains overvoltage)	Switch device off, switch it on again after 10 s
The display contrast is misadjusted	Use the knurled knob at the right side of the bottom to adjust the contrast.

The dosing unit is not filled correctly.

The reagent bottle is empty	Replace or top up reagent bottle
The hose is not immersed deeply enough in the reagent bottle	Immerse the hose deeper in the reagent bottle or top up reagents
The dosing unit is not properly locked	Latch dosing unit with a quarter rotation in the lower position

Air bubbles in the system

The hose is not immersed deeply enough in the reagent bottle	Immerse the hose deeper in the reagent bottle or top up reagents
The hose connections are not tight	Check: Has the hose been pulled out of the threaded connection? Fasten fingertight
	Replace hoses plus screw connectors
The valve is defective	Replace valve

When installing the dosing unit the piston rod is not pulled in correctly, the device “rattles”.

Oblique application of piston rod	Perform initial filling

The titration solution is not dosed.

The dosing unit is not properly filled	Perform initial filling
The dosing unit is not properly locked	Lock dosing unit with a quarter rotation in the lower position (🕮 **figure 51**)
The hose or the titration tip are bent or obstructed	Check proper flow through hose and tip, replace if required
The system contains air bubbles	Please refer to “Air bubbles in the dosing system”
Undissolved parts in the titration solution	Filter or replace titration solution
Valve hoses are mixed up	Connect hoses (🕮 **figure 5**)

Data transfer to the printer/computer does not work.

Set proper transfer parameters	Setting: 4800 baud, 7 data bits, no parity, no handshake
Using an appropriate cable	TZ 3090 for printer or TZ 3460 resp. TZ 3098 for PC
Cable correctly connected	The **middle** 4-channel socket is the RS-232-C interface 1
	Check cable connection; are the screws on the side of the PC tightened
	PC: Possibly „COM 1“ and „COM 2“ were mixed up
Defective cable	Replace cable
Malfunction of printer/PC or TitroLine KF	Switch device off, switch in on again after 10 s

For further information on titration disturbances, please refer to the "Karl-Fischer titration in practice" brochure.

10 Maintenance and care of the TitroLine *KF* Titrator

To maintain the functioning of the TitroLine *KF* Titrator certain inspection and maintenance work have to be carried out.

Note on wear and tear

In a larger sense, the valve and the glass cylinder are parts being subject to wear and tear; this means that their glass surfaces may wear down in use. This wear and tear may lead to an adulteration of the dosed volumes. For this reason, the dosing accuracy of the device should be checked.

Maintenance intervals

Normal operation:

➲ As a principle, all work has to be done at max. intervals of 3 months.

Under special load:

Special load is present, for instance, if solutions are used which are likely to attack glass, e.g. alkaline, fluoride-containing, phosphate-containing solutions, or if more than an average of 40 titrations are performed per day.

➲ Once per month the dosing unit has to be visually checked for damage.

➲ Once a year the inspection according to DIN 12 650, Part 6 or Part 7, or to ISO DIS 8655, Part 3, should be carried out, unless internal quality standards provide for different regulations.

➲ Once per quarter the electrical contacts (plugs, stirrer, manual key button) have to be checked for corrosion if the TitroLine *KF* Titrator is used in rooms the atmosphere of which occasionally contains corrosive matters.

In the case of disturbances:

➲ If any disturbance, malfunction, or another defect becomes obvious, maintenance work is required immediately.

➲ If one has to assume that ⚠ safe operation is impossible ⚠ ,
please observe chapter, “Warning and safety information“.

Maintenance work to be performed

➲ Check whether there is moisture below the dosing unit. To do so, remove the dosing unit (“Detaching and placement of the dosing unit” (chapter 6.6, **figure 48 to 51**). If moisture is actually present, one can
conclude that the piston in the cylinder is no longer tight.

➲ Check the hoses, screwed connections, and seals for visible damage, contamination and leakage, assembly of the screwed connections (🕮 detailed sketch of **figure 55**).

➲ Check the electrical plug connections for corrosion and physical damage.

➲ Defective parts have to be repaired or replaced by new parts. Defective glass parts have to be replaced in each case.

Interruptions of use

➲ If the TitroLine *KF* Titrator is not used for more than one week, the liquids contained in the system should be removed, and the TitroLine *K* Titrator is to be rinsed with alcohol. If this is omitted the piston may be destroyed, and the TitroLine *KF* Titrator will become untight. In addition, if the liquid is left within the system, one has to reckon with the occurrence of corrosion.

Cleaning

➲ The TitroLine *KF* Titrator should be cleaned using a damp cloth and normal household.

➲ The bottom and the rear panel have to be treated dry. In no case must liquid penetrate into the interior of the TitroLine *KF* Titrator.

11 Storage, transportation and environment

If the TitroLine *KF* Titrator has to be stored temporarily or transported, the original packing will provide the best protection for the device. In many case, however, this packing is no longer available, so that a comparable makeshift packing has to be put together. Sealing the TitroLine *KF* Titrator in a foil has proven to be a good solution.

Before doing so, please make sure that any KF reagent is removed from the dosing unit. Subsequently, you should rinse with alcohol (ethanol, isopropanol), subsequently, this alcohol has to be removed as well.

The device should be stored in a room meeting the following requirements:
- ➲ temperature between + 10 and + 40 °C for operation and storage,
- ➲ humidity according to EN 61 010, Part 1:
  max. ref. humidity 80 % for temperatures up to 31 °C,
  linear decrease down to 50 % relative humidity at a temperature of 40 °C

Your TitroLine *KF* Titrator will have a very long life, so that this information is in fact given far too early.

But when the end of its lifetime has come, please observe the regulations of your country and town regarding disposal.

12 Accessories and spare parts

Accessories

TZ 1052 drying oven for water determination according KF
TZ 1050 accessory set for TZ 1052 drying oven
TZ 1795 adapter are for solvent and waste bottle with S 40 thread
TZ 2004 bottle set for titration agent with 1 l brown-glass bottle
TZ 2005 bottle set-on unit with GL 45 thread for reagent bottles, e.g. Riedel-de Haën, Schott make
TZ 2008 bottle set-on unit with S 40 thread for reagent bottles, e.g. Merck make
TZ 2825 mini PC keyboard, splash-water resistant

TZ 3090 1.5 m data cable RS-232-C with plug for TZ 3460 printer connection:
1. side: 4-channel mini DIN plug
2. side: Western plug

TZ 3092 1.5 m data cable RS-232-C for the connection of Sartorius balances
1. side: 4-channel mini DIN plug
2. side: 25-channel plug (balance)

TZ 3093 1.5 m data cable RS-232-C for the connection of Mettler AT, PR, PM balances
1. side: 4-channel mini DIN plug
2. side: 15-channel plug (balance)

TZ 3099 1.5 m data cable RS-232-C for the connection of Mettler PG, AB-S, AG balances
1. side: 4-channel mini DIN plug
2. side: 9-channel plug (balance)

TZ 3094 1.0 m data cable RS-232-C: TitroLine *KF* →
TitroLine *KF* – Daisy Chain concept–
1. side and 2. side: 4-channel mini DIN plug

TZ 3095 1.5 m data cable RS-232-C with plug for printer connection (e.g. Epson LQ 300):
1. side: 4-channel mini DIN plug
2. side: 25-channel sub-Miniature-D plug
2. side: 9-channel sub-Miniature socket

TZ 3098 1.5 m data cable RS-232-C: 1. side: 4-channel mini DIN plug
2. side: 9-channel sub-Miniature-D socket

Adapter: 9-channel plug → 25-channel socket

TZ 3460 Printer with RS-232-C interface, included 1.5 m printer cable TZ 3090

Spare parts

TZ 1106 micro double platinum electrode
TZ 1770 titration vessel, complete (30 ... 150 ml)
TZ 1772 titration vessel, complete (80 ... 200 ml)
TZ 1773 septums (5 pieces) for titration vessel TZ 1770 and TZ 1772
TZ 1774 sealing set for TZ 1770 and TZ 1772 titration vessels
TZ 1775 glass vessel (30 ... 150 ml) for TZ 1770 KF titration vessel
TZ 1776 micro valve for TZ 1770 and TZ 1772 titration vessel
TZ 1778 micro valve (80 ... 200 ml) for TZ 1772 titration vessel
TZ 3000 valve
TZ 3130 dosing unit 20 ml, completely mounted
TZ 3630 mounting wrench

Notes:

Typ / Type / Type / Tipo: Titrator TitroLine *KF*

Bescheinigung des Herstellers

Wir bestätigen, dass das oben genannte Gerät gemäß DIN EN ISO 9001, Absatz 8.2.4 „Überwachung und Messung des Produkts“ geprüft wurde und dass die festgelegten Qualitätsanforderungen an das Produkt erfüllt werden.

Supplier’s Certificate

We certify that the equipment was verified according DIN EN ISO 9001, part 8.2.4 ”Monitoring and measurement of product” and that the specified requirements for the product are met.

Certificat du fournisseur

Nous certifions que le produit a été vérifié selon DIN EN ISO 9001, partie 8.2.4 « Surveillance et mesure du produit » et que les exigences spécifiées pour le produit sont respectées.

Certificado del fabricante

Hacemos constar que el equipo mencionado anteriormente ha sido probado de acuerdo con la norma DIN EN ISO 9001, sección 8.2.4 „Verificación y medición del producto“ y que se cumplen los requisitos especificados para el producto.

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